



# *Institute of Paper Science and Technology*

## **STATUS REPORTS**

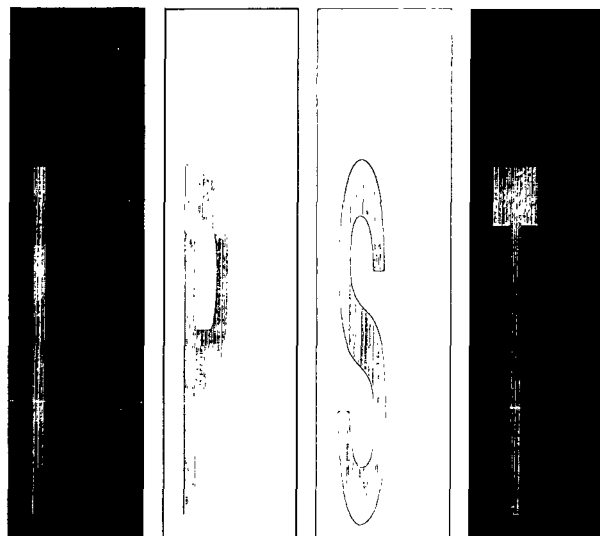
To The

**PAPER PHYSICS**

**PROJECT ADVISORY COMMITTEE**

April 4, 1991

Institute of Paper Science and Technology  
Atlanta, GA



*Atlanta, Georgia*



## INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

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# **STATUS REPORTS**

To The

**PAPER PHYSICS**

**PROJECT ADVISORY COMMITTEE**

April 4, 1991

Institute of Paper Science and Technology  
Atlanta, GA



March 15, 1991

TO: MEMBERS OF THE PAPER PHYSICS PROJECT ADVISORY COMMITTEE

Attached for your review are Status Reports for the projects to be reviewed and discussed at the Paper Physics Project Advisory Committee meeting scheduled for April 4-5, 1991, in Atlanta. A meeting agenda can be found inside the booklet.

Please note that the meeting is being held at the Wyndham Hotel located at 125 10th Street, one and one-half blocks east of the 10th Street (Midtown) MARTA Rail Station.

We look forward to seeing you on April 4-5. Best regards.

Sincerely yours,

Maclin S. Hall  
Paper Physics Group  
Engineering & Paper Materials Division

Attachment

*Institute of Paper Science and Technology, Inc.*

PAPER PHYSICS  
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## AGENDA

### PAPER PHYSICS PROJECT ADVISORY COMMITTEE

April 4-5, 1991  
Meeting at the Wyndham Hotel Midtown  
125 10th Street, Northeast  
Atlanta, Georgia

THURSDAY--April 4  
Woodruff Room

12:00	Lunch	
1:00	Welcome/Introductions IPST Antitrust Statement	Sethy/Hall
1:15	PROJECT REVIEWS	
	BOARD Board Properties and Performance Project 3571	Sarfarazi
	STRNG Strength Improvement and Failure Mechanisms Project 3469	Waterhouse
2:50	Break	
3:10	PROJECT REVIEWS	
	PPREL Process, Product, Properties Relationships Project 3467	Brodeur
	ONLIN On-Line Measurement of Paper Properties Project 3332	Hall

FRIDAY--April 5  
Crescent Room

8:00AM	Committee Discussion of Projects
12:00	Adjournment/Lunch



ON-LINE MEASUREMENT OF PAPER

STATUS REPORT

FOR

PROJECT 3332

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

April 4, 1991

### PROJECT SUMMARY

Project Title: ON-LINE MEASUREMENT OF PAPER PROPERTIES  
Project Code: ONLIN  
Project Number: 3332  
Division: Engineering and Paper Materials Division  
FY 90-91 Budget: \$70,000  
Project Staff: M. Hall, P. Brodeur, T. Jackson

#### PROJECT OBJECTIVE:

Shared support with DOE Project to develop sensors and instrumentation capable of measuring the velocity of ultrasound in the in-plane and thickness directions of paper while the paper web is moving at line speed.

#### PROGRAM AREAS:

Capital Effectiveness, End use Performance, Reduced Operating Costs

#### RATIONALE:

The ability to measure mechanical properties on the paper machine will provide a means to continuously monitor product quality related to end-use performance. The current objective is to extend the on-line measurement to include out-of-plane elastic constants which are needed for determination of several important mechanical properties. This project also provides data needed to relate product characteristics to process variables for paper machine control.

#### FY 90-91 GOALS:

- (1) Complete interface of integrated data collection system with ZD and in-plane transducers mounted on web handling system.
- (2) Check out and debug operation of data collection system on moving web.

- (3) Determine static performance of on-line sensors relative to laboratory instruments.
- (4) Determine dynamic performance of on-line sensors relative to their static performance.
- (5) Procure sample rolls of paper prepared with known processing conditions and selected process parameter changes.
- (6) Collect moving web data on rolls with known properties, determine response of instrumentation to selected process changes.
- (7) Issue annual progress report.

#### RESULTS LAST PERIOD: (April 1990 - November 1990)

- (1) The web handling system installation/startup was completed in June, 1990. This system is capable of speeds up to 2500 fpm, running in either an endless loop or a reel-to-reel mode. It is able to handle webs up to 14 inches in width as 30-foot endless loops or as reels on 3 inch cores up to 34 inches in diameter.
- (2) A new software package (WAVE) was purchased for use with the 386 computer. This package is designed for manipulating and analyzing digitized waveform data. This package has significantly increased the convenience and efficiency in preparing the software necessary to support the operation of the fluid-filled wheels and the collection of data.
- (3) A LeCroy 7200 Precision Digital oscilloscope, acquired in early October, 1990, provides the capability to capture a 50,000 point record length with one nanosecond time resolution. The time resolution is limited to 2.5 nanosecond in our present fluid-filled wheel configuration because the pulse record acquired spans approximately 100 microseconds.

- (4) Polycarbonate delay lines or "shims", about 12 mm long, positioned against each transducer in the fluid-filled wheels improve reflected pulse shapes. With improved pulse shapes we are using maximum peak detection instead of cross-correlation to minimize data processing time.
- (5) ZD measurements have been recorded with the fluid-filled wheels on moving webs in both closed loop and reel-to-reel modes. Pulse peak times are determined by the LeCroy 7200 and transferred to the 386 computer. WAVE software is used to calculate and display the caliper and sound velocity. Data is collected at 2 second intervals with this arrangement.
- (6) The "shims" described above also have a measurable pulse transit time that is sensitive to temperature. Monitoring the "shim" transit time can be used as a "thermometer" to correct for the effect of temperature changes in the wheels. This correction technique has been demonstrated with the above moving web measurements.
- (7) Work with in-plane measurements has been renewed with an evaluation of the variation of pulse-peak time differences in the CD shear and CD longitudinal modes. An acquisition system has been designed which will provide sequential measurement of the CD longitudinal, CD shear, and MD longitudinal signals with the LeCroy 7200 operating in the sequential mode.

#### GOALS FOR THIS PERIOD: (November 1990 - March 1991)

- (1) Improve transducer/"shim" configuration in wheels.
- (2) Refine data collection procedures; for ZD, for in-plane, and for both simultaneously.
- (3) Determine dynamic performance of sensors with representative paper samples and web speed conditions.
- (4) Draft progress report.

## RESULTS THIS PERIOD: (November 1990 - March 1991)

- (1) Special 1 Mhz transducers with a thin cap instead of the normal quarter wave cap were received and are now used with a 20 mm acrylic "shim" in one wheel.
- (2) Various ZD data collection options were investigated. It appears that averaging signals is necessary to compensate for variations around the tire. It also appears that pulse peak time measurements are not as repeatable as those determined by cross correlation.
- (3) ZD measurements on repeated reel runs.
- (4) In-plane acquisition circuit constructed and tested.
- (5) In-plane CD shear and MD longitudinal measurements on repeated reel runs.

## GOALS FOR NEXT PERIOD: (April 1991 - November 1991)

- (1) Determine dynamic performance of sensors with representative paper samples and web speed conditions.
- (2) Collect moving web data on sample rolls of paper prepared with known processing conditions and selected process changes.
- (3) Progress report.
- (4) Presentations to potential vendors.

## ON-LINE MEASUREMENT OF PAPER MECHANICAL PROPERTIES

The primary objective of this project is to develop sensors capable of measuring the velocity of ultrasound in the thickness and in-plane directions of paper while the paper web is moving at paper machine speed. The measurement of the velocity of ultrasound provides a nondestructive technique to characterize the mechanical properties of paper, allowing continuous monitoring of product quality. A further objective is to develop hardware and techniques using these sensors to control the paper manufacturing process. Successful implementation of the results of this project will provide more effective utilization of raw materials and energy while producing products with improved uniformity and quality. A major part of the support for this project is provided by funding from the U.S. Department of Energy, Contract No. DE-AC05-86CE40777.

### STATUS

Two approaches for making on-line measurement of ultrasonic velocity in the thickness direction (ZD) of paper have been developed. One uses modified, commercially available, fluid-filled wheel transducers, and the other uses IPST-made, elastomer-faced, PVDF wheel transducers. Our recent work has concentrated on using the fluid-filled wheels, because they can be used to simultaneously measure both out-of-plane ultrasound transit time and the caliper on moving paper webs. The paper web runs in the nip between two such wheels.

The transit time through the paper thickness is determined from a comparison of the arrival times of ultrasound pulses between the two transducers with and without the sample present. The caliper is determined from the difference in the arrival times of the first pulse and subsequent pulses that are reflected at the rubber-paper and transducer-water interfaces.

Bimorph or bender transducers are used for in-plane measurements. Three transducers are aligned in the surface of an aluminum drum to measure CD shear velocity and three others are positioned to measure MD longitudinal velocity.

A reel-to-reel or endless belt web handling system has been procured and installed. A stand for mounting and manipulating the wheel-transducers has also been procured and

installed. The drum with in-plane transducers is also mounted on the web handling system.

A LeCroy 7200 Precision Digital oscilloscope provides the capability to capture record lengths up to 50,000 points with one nanosecond time resolution. This together with a 386-type computer and a purchased software package (WAVE) provide the major components of the electronics to make ZD or in-plane measurements and collect data on moving webs.

## **WEB HANDLING SYSTEM AND TEST STAND**

An unwind/rewind web handling system was purchased and installation/startup completed in June, 1990. This web handling system includes a web guide and is capable of speeds up to 2500 fpm, running in either an endless loop or a reel-to-reel mode. A dancer arm provides adjustable tension in the loop mode, and tension is automatically controlled in the reel-to-reel mode. It is able to handle webs up to 14 inches in width with tension controllable from 0.5 to 4 pounds per linear inch. This system enables us to perform tests on 30-foot endless loops or reels on 3 inch cores up to 34 inches in diameter.

A portable test stand for mounting either the fluid-filled wheels or the IPST wheels was designed and constructed to operate with the unwind/rewind web handling system. It is portable in the sense that it can be disassembled from the web handling system and set up to operate as a C-frame on other web systems.

This test stand has cross web positioning in one inch increments. The wheel mounts are extended and retracted by air cylinders. Motors are provided to match wheel-to-web speed before closing the wheels onto the web. The wheel axle spacing (nip pressure) is adjustable. We have found that the force provided by the air cylinders is not sufficient to maintain a fixed axle distance with "stiff" or out-of-round tires. A Kaman displacement sensor may be used to monitor axle distance variation. For some experiments the wheels are blocked in the closed position.

## FLUID-FILLED WHEEL TRANSDUCERS

Work continues with modified commercial, fluid-filled wheels to make out-of-plane ultrasound velocity measurements and caliper measurements on moving paper webs. Here a transducer is mounted to a rigid frame which is encapsulated in a fluid-filled, rubber wheel. The paper runs in the nip between two such wheels, and the caliper and velocity of ultrasound in the thickness direction or ZD of paper are calculated from the differences in the transit times of ultrasonic pulses between the two transducers with and without the sample present. By using the pulses reflected at the rubber-paper and transducer-fluid interfaces in addition to the direct pulse, the caliper of the paper can be determined.

Under static conditions, values of caliper and velocity determined with the fluid-filled wheels are in good agreement with measurements made with laboratory instruments. This has been demonstrated with a set of 29 samples which include a variety of papers and calipers.

We observed that interference of the signals reflected at the liquid-rubber and rubber-paper interfaces affected the pulse shapes and caused errors in the determination of pulse time differences. Experiments with added rubber thickness, indicated that these signals could be separated by using thicker "tires" on the fluid-filled wheels. New wheels with thicker tires were purchased, providing significant improvement in pulse shapes.

With the ability to more accurately capture and analyze pulses and their time difference, we observed that the reflected pulse shape affected the time difference determination. We attributed this to the reflection of the pulse at the transducer surface. The standard immersion transducers are made with a one quarter wavelength plastic cover. We have purchased special transducers made with a cover of the minimum thickness needed for a water seal, much thinner than the conventional quarter wave cover.

While the special transducers were on order, we experimented with several techniques to provide a suitable reflective surface. We found that polycarbonate or acrylic rods positioned against each transducer gives "clean" reflected pulses. The length of these delay lines or "shims" and the spacing of the transducers in the wheels must be carefully selected to minimize overlap of the reflected pulses.



We reported previously that temperature changes in the wheels encountered with moving-web operation change the velocity of sound in the wheels and affect the pulse delay time measurements. In order to monitor and correct for this, we mounted a thermocouple in one of the wheels as part of a digital thermometer that was interfaced to the computer over an GPIB bus. We found it difficult to eliminate a high frequency noise and obtain sufficient temperature resolution with the instrument on hand and a more sophisticated instrument would be required for temperature measurement. Fortunately, the "shims" described above also have a measurable pulse transit time that is sensitive to temperature. Monitoring the "shim" transit time can be used as a "thermometer" to correct for the effect of temperature changes in the wheels.

We have two sets of "thick" (3/16 inch) tires. One set is made from soft rubber and the other is made from a harder polyurethane. There are advantages and disadvantages for each type. The "soft" tires are similar to the soft rubber that has been used for soft platen caliper and for the transducers used for laboratory ZD measurements. Soft rubber is believed to be desirable for coupling ultrasound to paper for longitudinal ZD measurements. However, the soft tire distorts as speed is increased. Our first set was scuffed by rubbing against the wheel support when the motor driven wheel speeds were first tested. We have new tires and have modified the wheel mounts to provide more space for tire distortion.

The "hard" tires are quite stiff and any out-of-roundness will cause a variation in pressure on the paper being measured and make it more difficult to hold the distance between wheel axles fixed. On the other hand, the hard tires do provide good signal strength, do not distort at higher speeds, and would be expected to wear better in use. A choice between the use of the "hard" or the "soft" tires is not yet clear.

#### DATA COLLECTION WITH FLUID-FILLED WHEELS

A software package (WAVE) was purchased for use with the 386-type computer. This package is specially designed for manipulating and analyzing digitized waveform data. This package has significantly increased the convenience and efficiency in preparing the software necessary to support the operation of the fluid-filled wheels and the collection of data.

We reviewed the speed and capacity of our electronics instrumentation. We had been using a Tektronix model 2432 digitizing oscilloscope which has a record length of 1024 per channel and performs well when the task is to record only one pulse. In order to determine caliper with the fluid-filled wheels, one must record a three-pulse signal extending over more than 100 microseconds. To obtain sufficient resolution for accurate time measurement it is necessary to record the data at three separate times, once for each pulse location. Although this takes more time, it is adequate for static measurements. However, for moving web operation the measurement made at different times would be at different web locations and give questionable results. Therefore, the capability to capture a longer record length at a faster digitizing rate was needed to simultaneously capture all of the pulses of interest with sufficient time resolution. A LeCroy 7200 Precision Digital oscilloscope was acquired in early October, 1990, to provide this capability.

The arrangement of the electronics for ZD data collection is shown in Figure 1. The travel of the ultrasonic pulses and the formulas used to determine caliper and transit time in the paper are shown in Figure 2. A typical received signal is shown at low resolution in Figure 3.

The LeCroy 7200 provides a number of on-board data collection and analysis options. We have experimented with a variety of combinations looking for a desirable compromise between measurement repeatability and sampling rate. Figures 4a and 4b show an example of a repeated run of a 3200 foot reel made up of several sections of 42 lb liner spliced together from different rolls. This data was obtained by first recording the  $t_1$  reference with the tires rotating, no sample, and averaging 1000 signals. The tires are then moved onto the web and the  $t_1$  pulse produced by averaging 500 signals is sent to the 386 computer and cross correlated with the  $t_1$  reference. This takes approximately 4 seconds. The data presented in Figures 4a and 4b was taken with the web moving at about 50 feet per minute.

The "soft" tire set, containing the 1 Mhz, "thin cap" transducers with a 20 mm acrylic shim in one wheel was used.

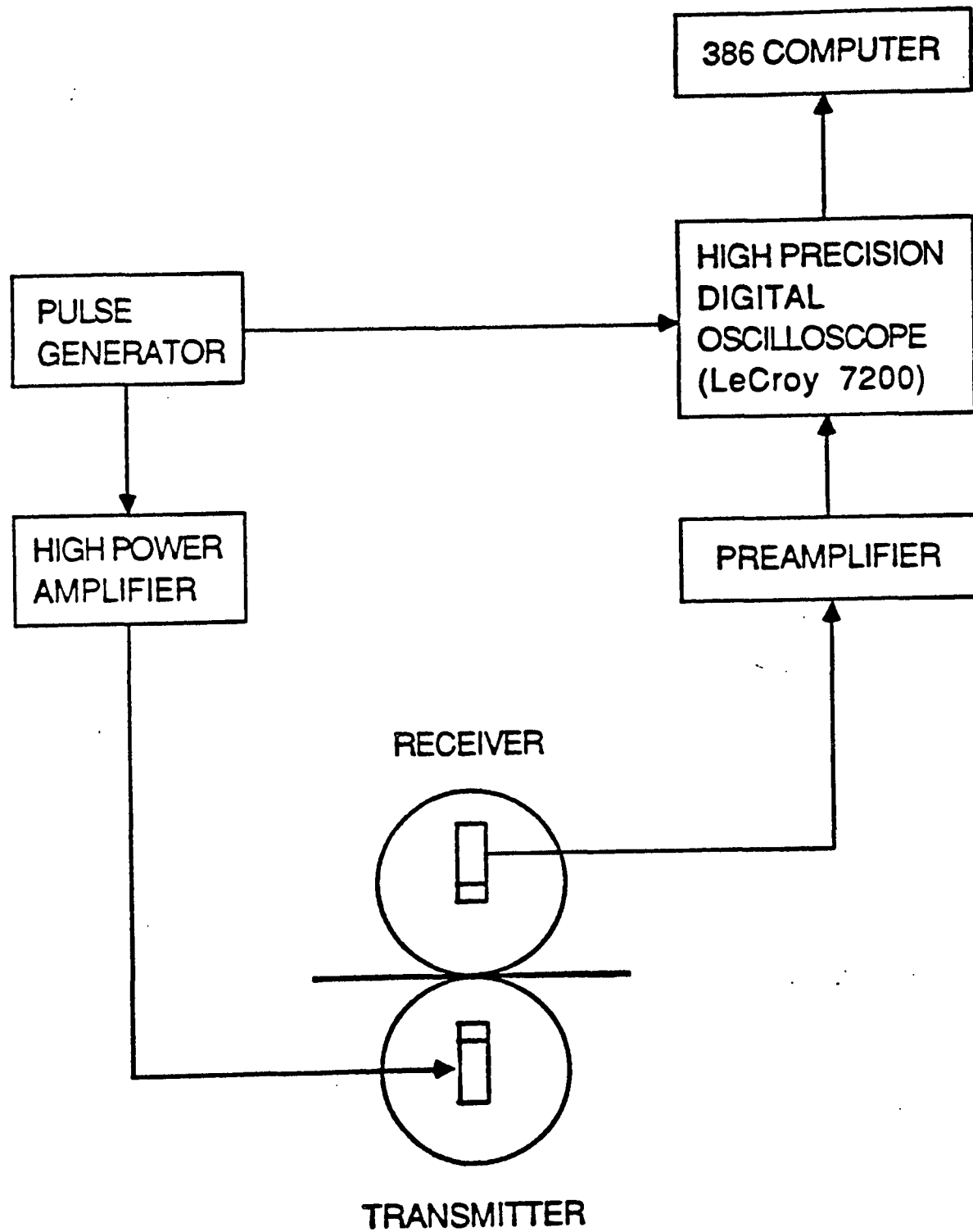
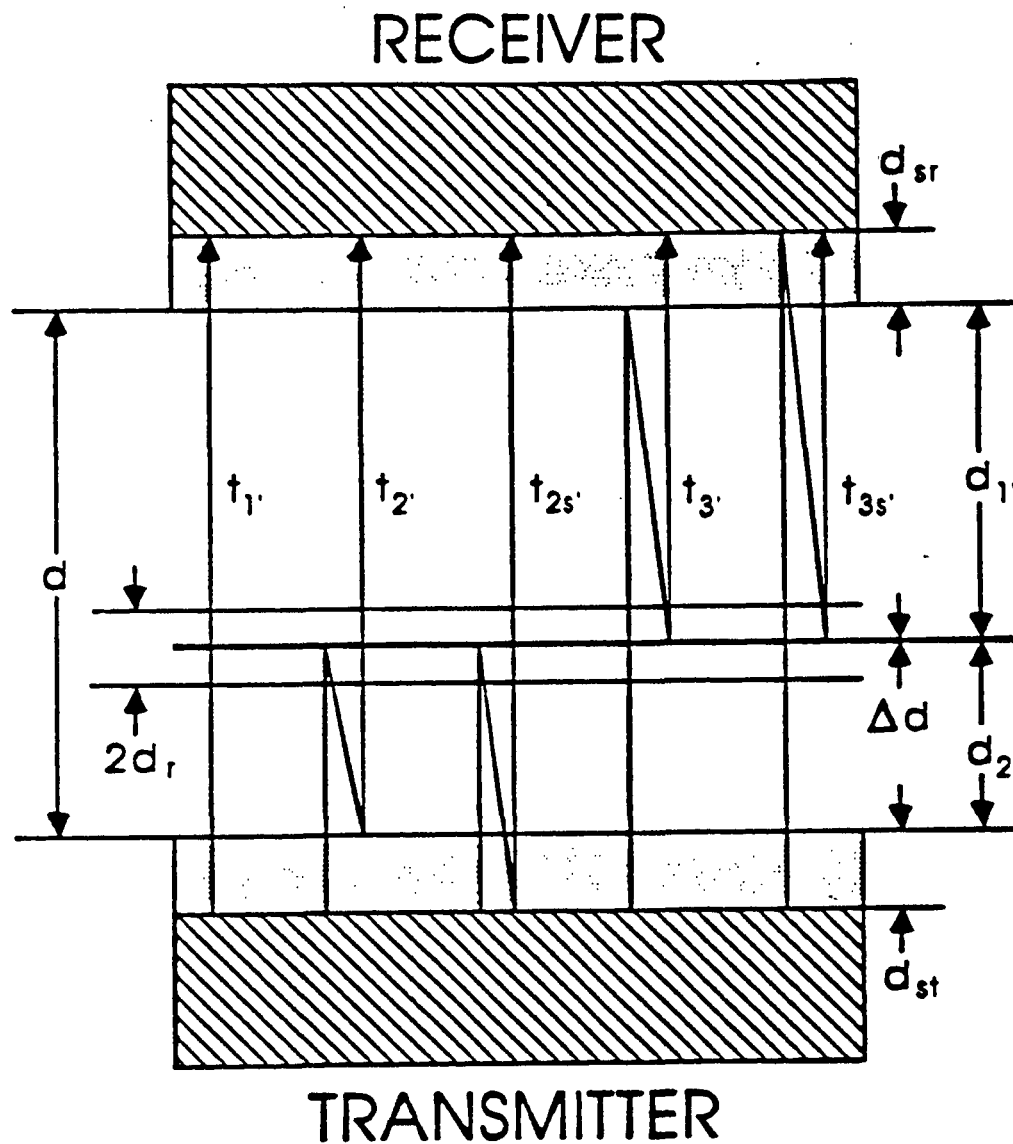


Figure 1.



Temperature Dependent Caliper and Transit Time:

$$\Delta d = kv_f(T=T^0) \delta t_{avg}(T=T^0) [1 - \delta t'_{avg}(T) / \delta t_{avg}(\Delta t_s)]$$

$$\Delta t = [t_1(T) - t_1(\Delta t_s)] + [\delta t_{avg}(\Delta t_s) - \delta t'_{avg}(T)]$$

Figure 2.

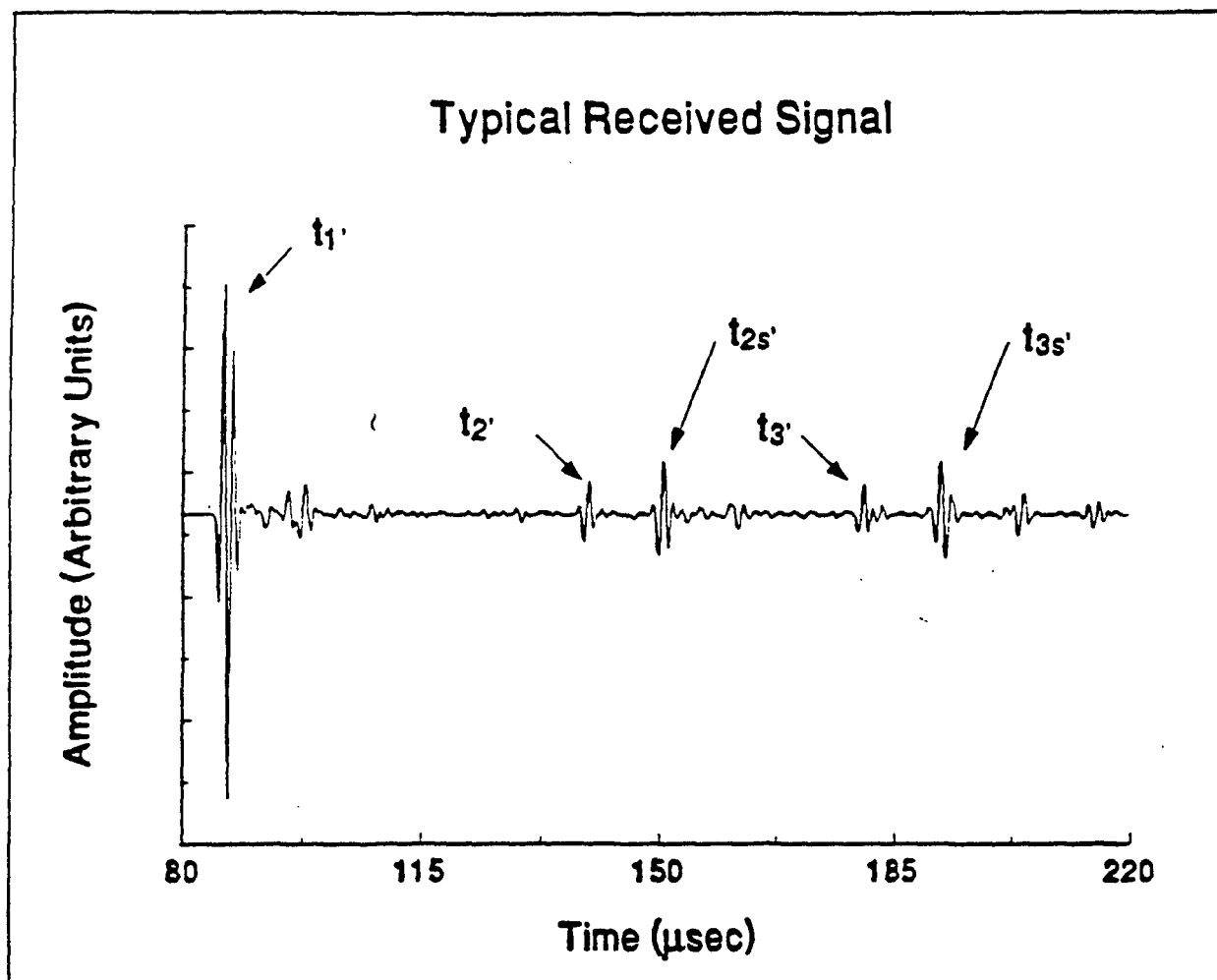


Figure 3.

03\_06\_91 -&gt; 42lb Reel to Reel

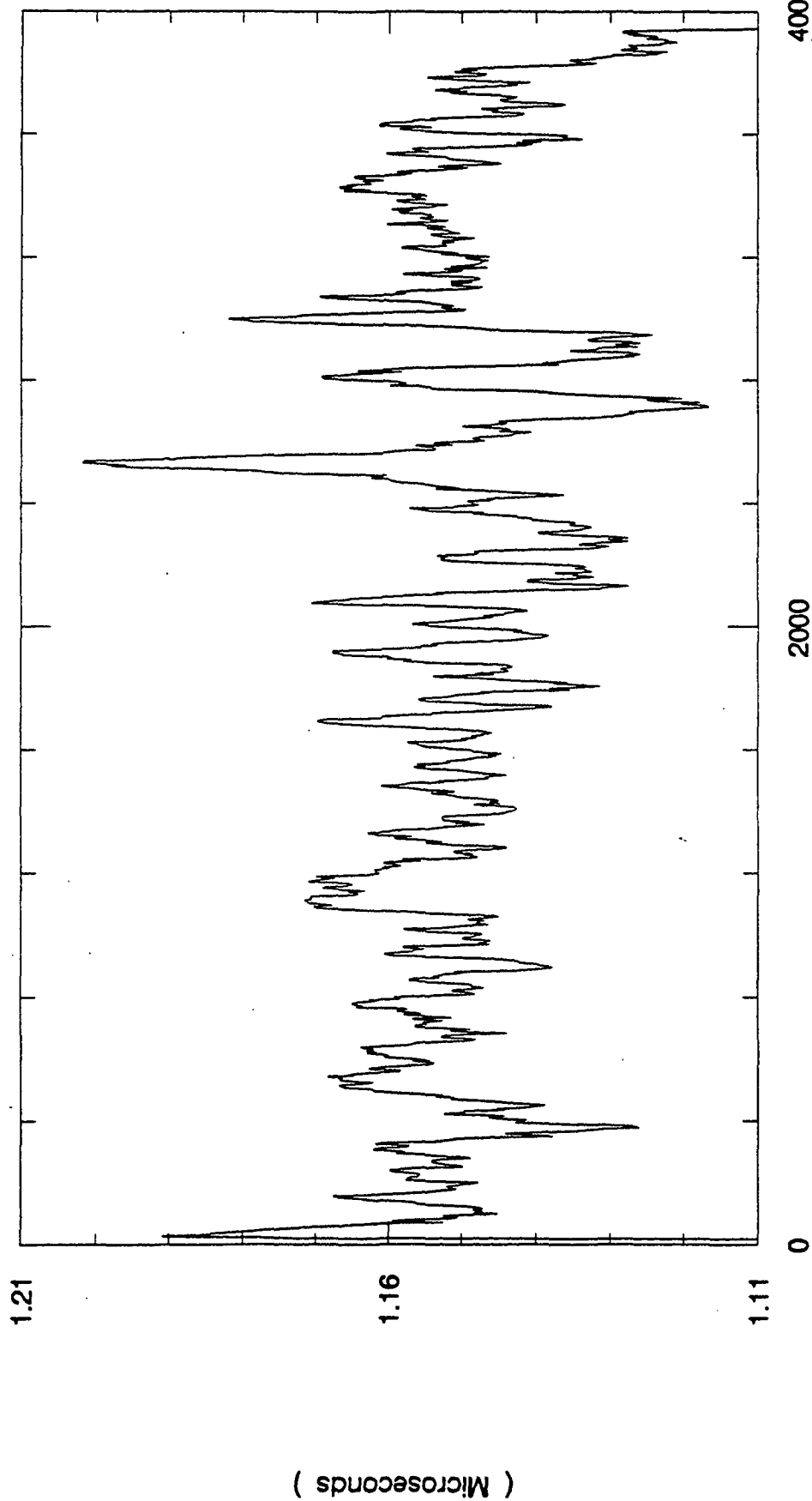
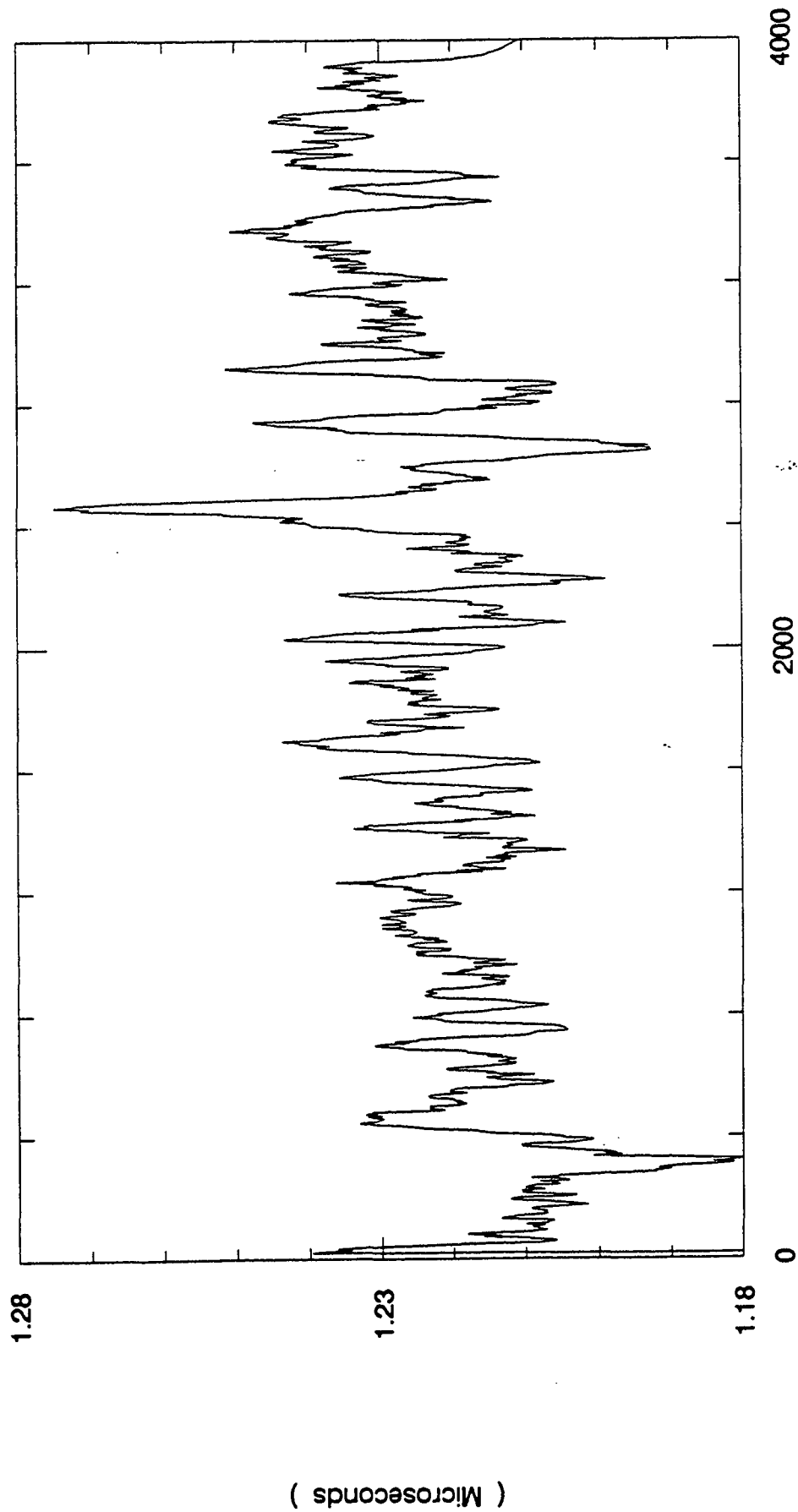


Figure 4a.

03\_07\_1 -&gt; 42lb Reel to Reel



smooth11( corr( ref\_1, t\_1 ) ) ( Seconds )

Figure 4b.

## IN-PLANE BIMORPH TRANSDUCERS

For in-plane measurements, we are using bimorph or bender transducers similar to those developed for in-plane measurements in the laboratory instruments. A technique has been developed to adhere a metal wear surface to the tip of the bimorph transducer to provide a more durable contacting surface. These offer potential advantages over transducers used in previous on-line, in-plane work in that they are broadbanded and have a much smaller area of contact with the web.

An aluminum drum or roll has been built to house the transducers in special spring-loaded fixtures to minimize the variability in the loading of the transducers to the web. The drum surface has a grooved pattern designed to achieve strong, narrow acoustic pulses in the plane of the paper. Provision is made in the web handling system described above to mount this drum.

Figure 5 shows a schematic of the data acquisition system interfacing the Lecroy 7200 to the drum in which the bender transducers are mounted. We are using three transducers aligned in the cross direction (CD), the transmitter on one end, the first receiver spaced at 4.6 cm and the second at an additional 8.2 cm. These transducers are oriented for the CD shear mode. Three transducers are aligned in the machine direction (MD), the transmitter on one end, the first receiver spaced at 6.6 cm and the second at an additional 10.1 cm. The transducers are oriented for the MD longitudinal mode. In this configuration the near receiver for the CD shear mode is used as the far receiver for the MD longitudinal mode.

The LeCroy 7200 is used in a sequencing mode and the time difference between the first peak of the received pulses is determined. Both CD shear and MD longitudinal velocities are measured each revolution of the drum. CD shear velocity data recorded for a 1500 foot section of a reel of 42 lb liner are shown in Figure 6a and a repeat run of the same section are shown in Figure 6b. The MD longitudinal velocity data taken for these runs are shown in Figures 7a and 7b.



## In-plane Acquisition System Schematic

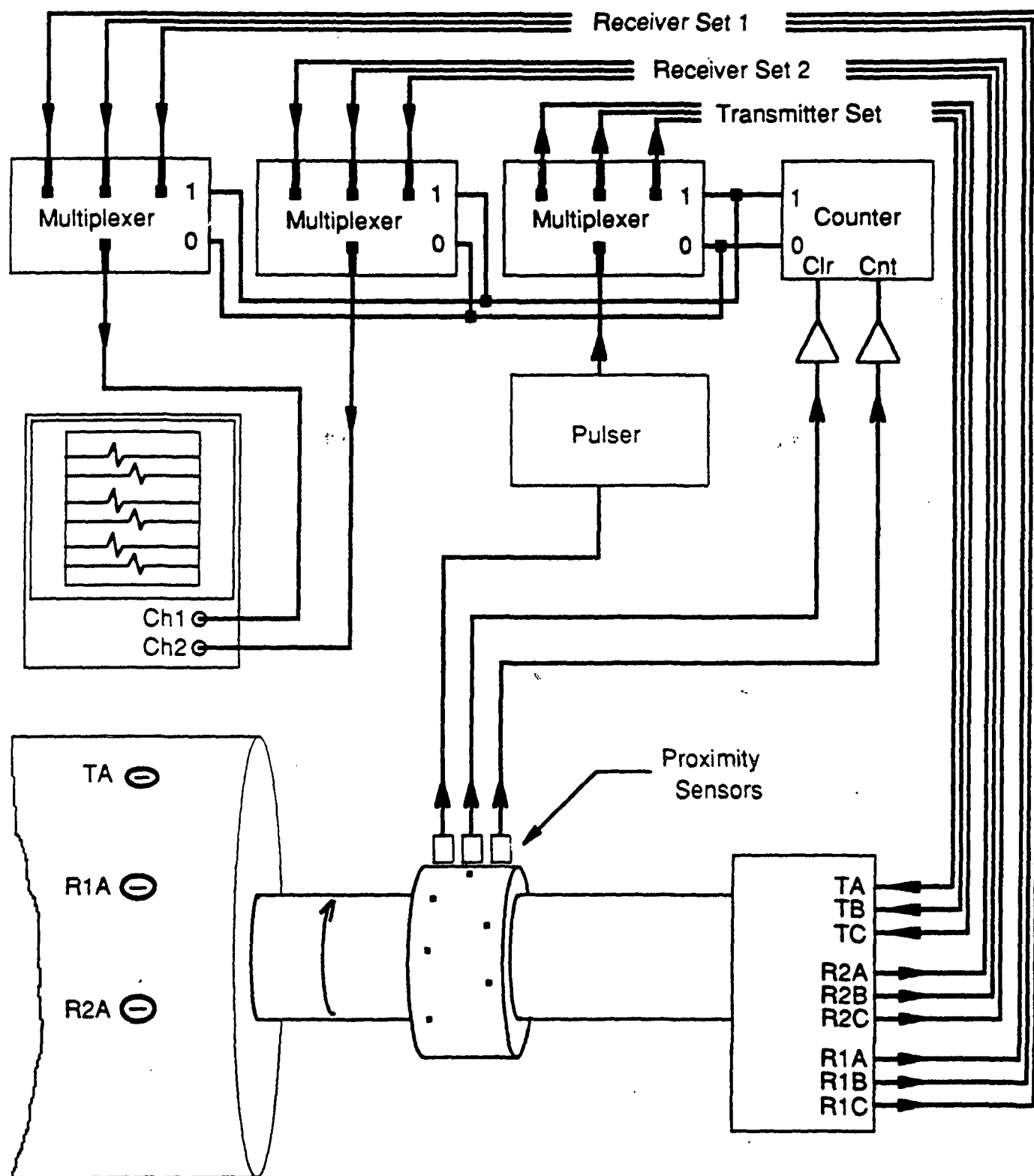


Figure 5.

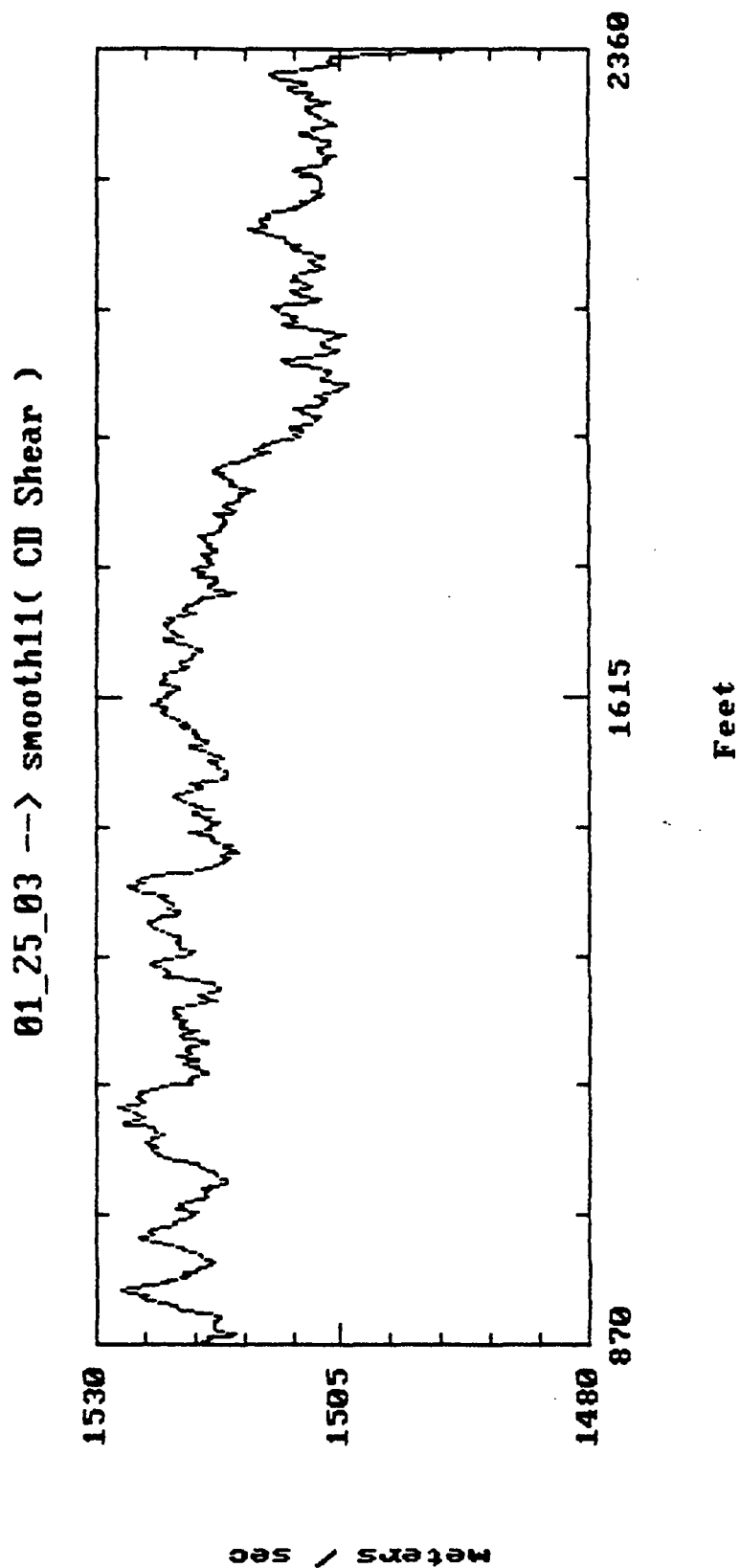


Figure 6a.

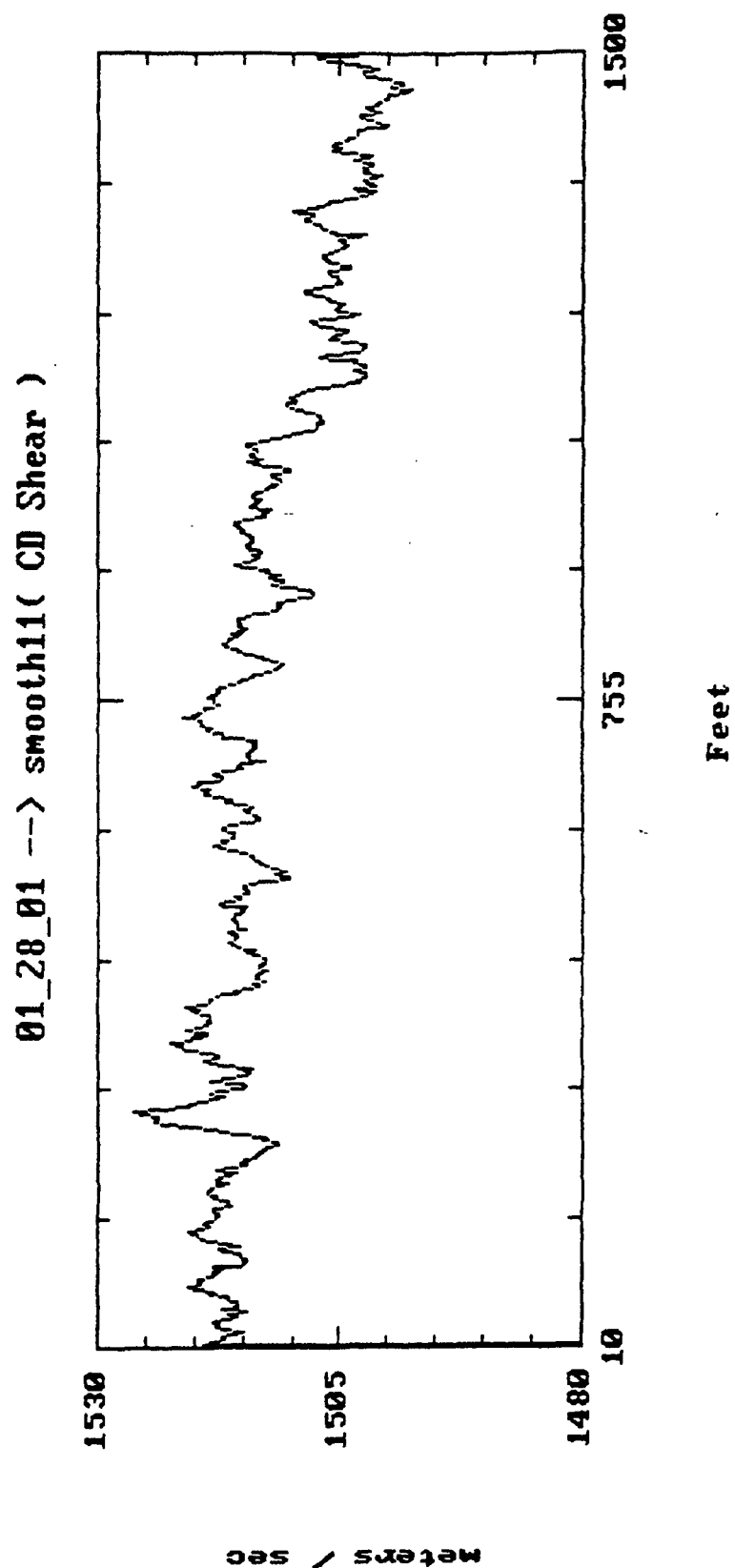


Figure 6b.

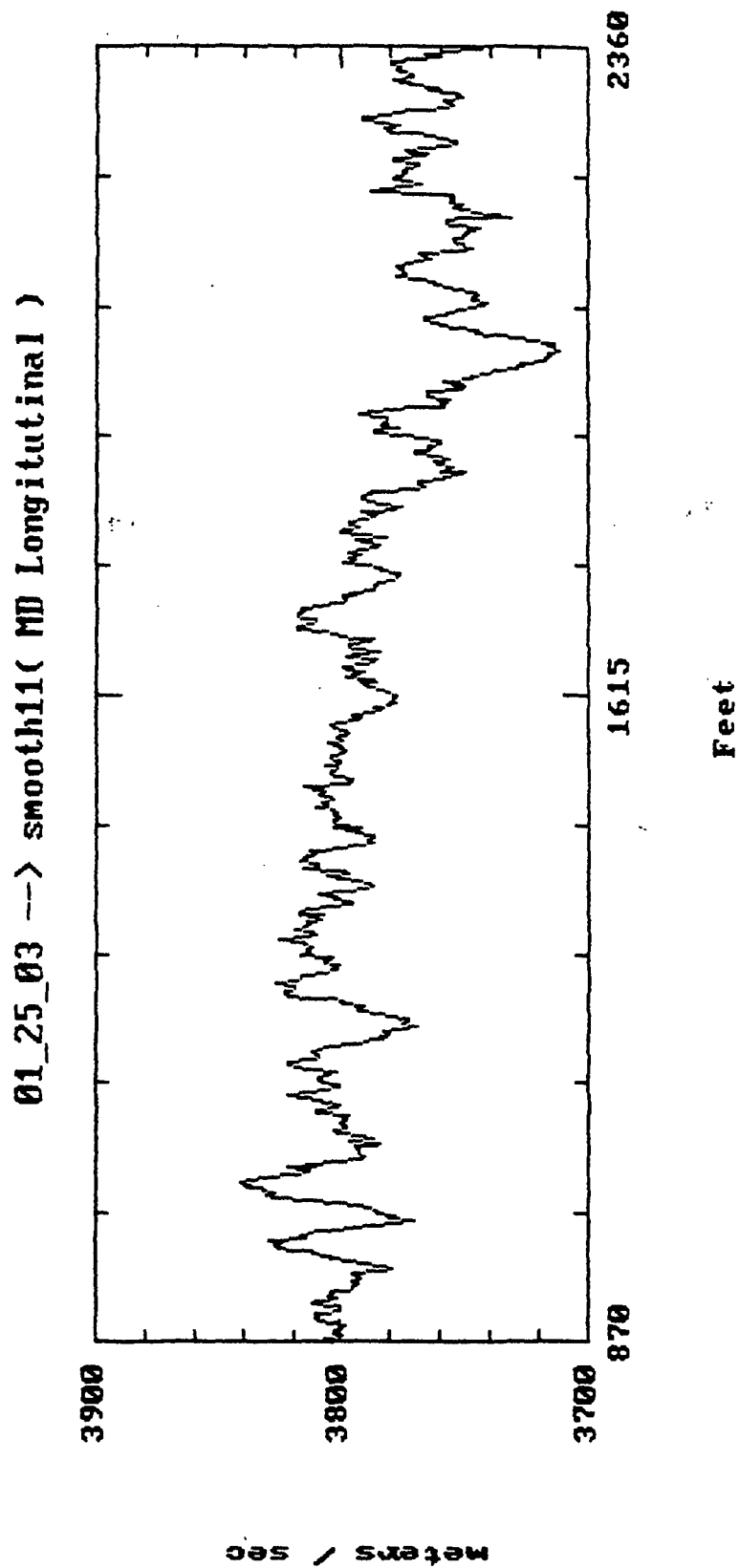


Figure 7a.

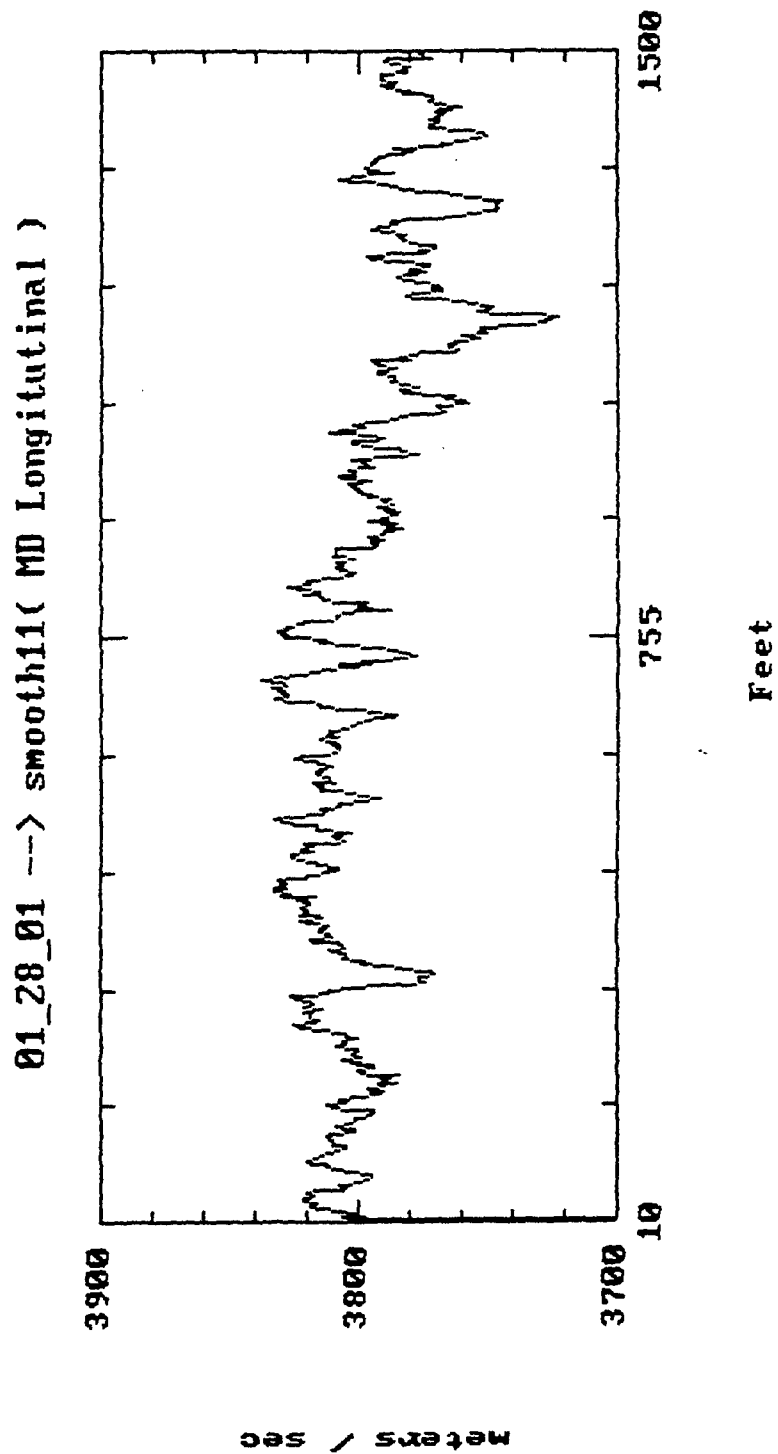


Figure 7b.

PROCESS, PRODUCT, PROPERTIES RELATIONSHIPS

STATUS REPORT

FOR

PROJECT 3467

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

April 4, 1991

## PROJECT SUMMARY

Project Title: PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS  
Project Code: PPREL  
Project Number: 3467  
Division: Engineering and Paper Materials Division  
FY 90-91 Budget: \$200,000  
Project Staff: P. Brodeur, M. Hall, T. Jackson

PROJECT OBJECTIVE: Develop relationships between critical physical properties of paper and board materials and the selection of raw materials, the principles of sheet design, processing conditions, and end-use performance.

PROGRAM AREAS: End Use Performance

RATIONALE: It is important to understand the relationships between process variables, end-use performance, and paper and board properties in order to improve these products or maintain performance within close tolerances while effectively utilizing available raw materials, minimizing energy requirements, and minimizing environmental impacts.

## FY 90-91 GOALS:

- (1) Non-destructive Ultrasonic Evaluation of Paper Materials
  - Improve accuracy of static measurements in the fluid-filled wheels setup.
  - Investigate amplitude and frequency content of ZD ultrasonic measurements (Fourier analysis) in order to understand sound dispersion and attenuation in paper.
  - Develop a technique to evaluate temperature and moisture effects on ZD paper elastic properties.
  - Prepare and submit two papers about static ZD measurements (one in a technical review and one in a scientific journal).
- (2) Non-destructive Microwave Evaluation of Paper Materials
  - Establish a joint research project IPST - Georgia Tech Research Institute (GTRI) to develop an on-line microwave instrument for fiber orientation measurement.

(3) Plasma Processing of Paper Materials

- Prepare an exploratory research proposal about plasma-assisted etching of paper and internal stress evaluation.

(4) Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

- Complete and submit for publication a paper about the motion of fluid suspended fibers in a standing wave field.
- Investigate techniques to align fibers in a slow moving suspension using ultrasonic standing waves.

(5) Acoustic Wave Propagation Through Porous Media

- Develop an experimental setup to perform acoustic velocity and attenuation measurements through porous media as a function of temperature, frequency, and pore size. The main goal is to examine the relationships between ultrasonic measurements and anomalous specific heat measurement of vicinal water (in collaboration with F. Etzler).

RESULTS LAST PERIOD: (APRIL 1990 - DECEMBER 1990)

(1) Non-destructive Ultrasonic Evaluation of Paper Materials

- A water tank has been built to study wave propagation in various materials and to simulate wave propagation in the ZD fluid-filled wheels (FFW) setup.
- Water tank studies have shown that some of the dispersion observed between transmitted pulses and transducer-water interface reflection pulses in the FFW setup is due to the quarter wave plate epoxy layer in front of each transducer. This problem has been solved by adding plastic reflectors in front of the transducers. Special immersion transducers with thin protective layers have been ordered.
- A technique has been developed based on maximum peak amplitude detection to evaluate pulse travelling times in the FFW setup. Although not as accurate as cross-correlation analysis, the method provides fast measurements with limited raw data.
- Using the FFW setup and hard-rubber tires (molded urethane wheels), caliper and phase velocity measurements have been collected for 29 paper samples from 50 to 1800 micrometers. Tested materials are fine (thin) papers, linerboards, mediums and heavy weight boards.
- FFW caliper agrees very well with standard soft-platen caliper over the full caliper range. This is an indication that applied pressure on paper remains constant even though caliper is not constant.



- FFW caliper calibration using soft-platen caliper allows accurate determination of the composite fluid velocity (fluid + rubber) in the wheels. The measured velocity does not agree with the predicted velocity. Hence, a calibration factor is found. Since caliper evaluation is pressure sensitive, the calibration factor takes into account pressure effects. More work is required to confirm the result.
- FFW velocity is in good agreement with the standard laboratory out-of-plane velocity, except for fine papers and medium materials. More work is underway to understand the discrepancies.
- Using frequency spectrum analysis techniques, it is shown that as the basis weight is increased, transmitted signals through paper are dispersed toward lower frequencies. This is in agreement with sound attenuation effects in dispersive materials.
- A study has been undertaken to evaluate phase and group velocities as a function of frequency in the paper thickness direction. Reference and sample pulses obtained from the fluid-filled wheels setup are used for that purpose. Preliminary measurements are available in the frequency range 0 to 1.5 MHz.
- Kramers-Kronig relationships have been used to predict sound attenuation in paper from dispersion measurements. Deduced attenuation is in good agreement with measured attenuation for some of the 29 samples available.
- Comparison of the deduced and measured attenuation coefficients as a function of frequency allows determination of the reflection coefficient at the paper-rubber interface. Work is in progress to understand the meaning of the preliminary results and their relationships to paper properties.
- A pulser-receiver has been purchased to perform pulse-echo measurements in paper thickness direction. It is hoped that pulse-echo measurements will provide direct evaluation of the reflection coefficient. Preliminary measurements with hard-rubber tires are not conclusive.
- Initially, it was intended to use the water tank to determine relationships between out-of-plane longitudinal sound velocity measurements and temperature and moisture effects (samples sealed in a waterproof holders). Due to poor acoustic coupling in sample holders, the method has been discarded. Instead, it is planned to perform the experiment in a humidity chamber using a duplicate of the FFW setup.
- Abstracts have been submitted to "Ultrasonics International 91" and the "1991 International Paper Physics Conference".

(2) Non-destructive Microwave Evaluation of Paper Materials

- Preliminary work has been undertaken in collaboration with the Georgia Tech Research Institute to develop a microwave technique to measure fiber orientation.

- An exploratory research proposal has been completed by G. Hopkins from GTRI: "Development of a Microwave Dielectric Anisotropy Measurement Technique for On-line Determination of Paper Fiber Orientation". The proposed technique is based on electronic polarization rotation in a waveguide.

(3) Plasma Processing of Paper Materials

- A390 problem was completed by T. Schwantes: "Internal Investigation of Elastic Properties of Paper Using Non-mechanical Layer Removal Techniques".
- An exploratory research proposal has been written: "Investigation of Paper Properties using Microwave Plasma Etching". It concerns the investigation of paper internal properties using microwave plasma etching. Principal investigators are: P. Brodeur (IPST), J. Waterhouse (IPST), and M. Wertheimer (Ecole Polytechnique de Montreal).

(4) Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

- A paper entitled "Motion of Fluid Suspended Fibers in a Standing Wave field" has been accepted for publication in Ultrasonics. The paper presents a model aimed at predicting acoustic fiber layering and reorientation effects in a standing wave field.

GOALS FOR THIS PERIOD: (DECEMBER 1990 - APRIL 1991)

(1) Non-destructive Evaluation of Paper Materials

- Investigate amplitude and frequency content of ZD ultrasonic measurements (Fourier analysis) in order to understand sound dispersion and attenuation effects in paper.
- Verify Kramers-Kronig relationships for paper.
- Investigate reflection coefficient.
- Develop a technique to determine temperature and moisture effects on ZD paper elastic properties.
- Prepare and submit one paper about static ZD measurements.

(2) Non-destructive Microwave Evaluation of Paper Materials

- Establish a joint research project IPST - Georgia Tech Research Institute (GTRI) to develop an on-line microwave instrument for fiber orientation measurement.

(3) Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

- Investigate techniques to align fibers in a slow moving suspension using ultrasonic standing waves.

## RESULTS THIS PERIOD: (DECEMBER 1990 - APRIL 1991)

## (1) Non-destructive Evaluation of Paper Materials

- A duplicate of the fluid-filled wheels setup has been built to perform caliper and velocity measurements in a controlled temperature/humidity chamber.
- A paper describing the FFW technique has been accepted for oral presentation at the Ultrasonics International 91 Conference and Exhibition, Le Touquet, France, July 1-4.
- A paper on sound dispersion and attenuation in paper has been accepted for oral presentation at the 1991 International Paper Physics Conference, Hawaii, September 22-27.

## (2) Non-destructive Microwave Evaluation of Paper Materials

- A revised exploratory proposal on microwave fiber orientation analysis has been prepared by GTRI. It is now proposed to do free-space measurements at much higher frequency than previously anticipated.

## (3) Plasma Processing of Paper Materials

- The exploratory research proposal has been updated to take into account etching times. Three specially selected linerboard samples has been received and preliminary characterization has begun.

## (3) Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

- A technique has been proposed to align fibers in a small flow tube.
- Preliminary work has been undertaken on the design of custom-made piezoelectric ceramic transducers to achieve fiber alignment.

## GOALS FOR NEXT PERIOD: (APRIL 1991 - OCTOBER 1991)

## (1) Non-destructive Ultrasonic Evaluation of Paper Materials

- Improve accuracy of static measurements in the fluid-filled wheels setup.
- Investigate amplitude and frequency content of ZD ultrasonic measurements (Fourier analysis) in order to understand sound dispersion and attenuation in paper.
- Begin experiments on ZD paper elastic properties in a controlled temperature/humidity chamber.
- Prepare two oral communications at conferences.

- Prepare two articles on FFW static measurements and sound dispersion and attenuation in paper.
- (2) Non-destructive Microwave Evaluation of Paper Materials
- Perform exploratory fiber orientation microwave measurements at GTRI and compare results to in-plane ultrasonic maximum stiffness lean angle.
- (3) Plasma Processing of Paper Materials
- Perform plasma-assisted etching experiments and evaluate thickness direction internal stress distribution in linerboard materials.
- (4) Acoustic Radiation Pressure Effects on Fluid Suspended Fibers
- Prepare an exploratory research proposal on acoustic fiber alignment in a small flow tube.

PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS  
PROJECT 3467

## A. Introduction

The main objective of Project 3467 is to study physical properties of paper and board materials and develop relationships with raw materials, principles of sheet design, processing conditions, and end-use performance. During the past years, non-destructive ultrasonic characterization of paper elastic properties has been the major research interest within Project 3467. Hence, it is not surprising that a great deal of effort during the March 1990 - March 1991 period continued to be devoted to ultrasonic characterization. However, new exploratory research interests have been developed during this period to enlarge the scope of activities. As a result, the research program has been reshaped into four research areas. They are now briefly introduced:

1. Non-destructive Ultrasonic Evaluation of Paper Materials

This research area encompasses the non-destructive investigation of out-of-plane and in-plane elastic properties of paper. This includes improvement and/or development of new ultrasonic sensing and analysis techniques. Priority in this matter is aimed at supporting Project 3332 (On-line Measurements of Mechanical Properties). In the physics-related sub-area, frequency domain analysis of ultrasonic pulses launched in the out-of-plane direction has evolved toward the fundamental analysis of sound dispersion and attenuation in paper materials. Work has been undertaken to evaluate temperature and moisture effects on ZD ultrasonic measurements.

2. Non-destructive Microwave Evaluation of Paper Materials

Development of this program has been generated from the interest in non-destructive, non-contact, moisture insensitive, and density variation independent fiber orientation determination. Availability of free-space microwave-based fiber orientation measurements would fulfill two

objectives. The first one concerns effects of drying-induced stresses on in-plane elastic properties of paper. It is expected that these effects could be evaluated from the comparative analysis of in-plane maximum stiffness lean angle and fiber orientation angle. The second objective implies the development of an on-line fiber orientation sensor to be used in process control.

### 3. Plasma Processing of Paper Materials

Plasma processing can be defined as the plasma-induced surface chemical treatment of materials. This is a wide topic which encompasses foreign material deposition, surface modification and etching. Our interest here concerns plasma-assisted etching as a new non-mechanical layer removal technique to study paper internal properties. It is believed that thickness direction paper internal stress distribution, as obtained from residual stress analysis, could be determined more accurately.

### 4. Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

This last topic is indirectly related to paper. In fact, it is aimed toward the analysis of acoustic radiation pressure effects on water-suspended cellulosic fibers. More specifically, it involves the development of a new ultrasonic technique to reposition and/or reorient fibers suspended in water. Among interests for the technique is the possibility of aligning (and straightening) fibers along a preferential direction. This technique, implemented with laser scanning of water-diluted slow moving fibers in a small tube, could lead to the development of a novel fiber analyzer system. Such a system would allow accurate determination of fiber width (coarseness) distribution. Moreover, if multiple scanning could be achieved on individual fibers, coarseness profile along the fiber length and fiber length distribution could be determined as well. If successful, this technique could also be used to evaluate curl and/or stiffness of individual fibers.

## B. Research Program

### 1. Non-destructive Ultrasonic Evaluation of Paper Materials

#### a. Out-of-plane FFW Set-up Static Measurements

The so-called fluid-filled wheels (FFW) setup has been developed for on-line sensing of paper out-of-plane elastic properties (Project 3332). In order to successfully achieve this goal, a full understanding of the instrument's capabilities in a static mode needs to be achieved. Hence, considerable attention has been drawn to this instrument during the past year as part of Project 3467. While measurement principles and results (static results only) are presented here, hardware, software and signal processing improvements are reported in Project 3332.

#### Principles: Temperature Independent Measurements

Principles have been reported previously. However, major improvements in the measurement techniques have led to a new and hopefully more definitive reformulation of the principles. Consider the transmission mode arrangement shown in Figure 1. Two identical immersion piezoelectric ceramic transducers are used to launch (transmitter T) and receive (receiver R) ultrasonic pulses. A plastic delay line located in front of the transmitter is used to monitor temperature variations in the wheels as discussed later on. The transmitter-delay line assembly and the receiver are separated by a fixed distance  $d$ . Water-filled rubber wheels are used to provide adequate acoustic coupling between transducers and paper specimens introduced in the nip. Assuming that wheels are under mechanical pressure to minimize the air gap thickness when no paper is present, the separation distances between the air-rubber interface and transducers T and R are defined by  $d_s + d_1$  and  $d_2$ , respectively. Thus,

$$d = d_s + d_1 + d_2 \quad (1)$$

Defining paper thickness (caliper) as  $\Delta d$ , a similar equation applies when paper is in the nip,

$$d = d_s + d_1' + d_2' + \Delta d \quad (2)$$

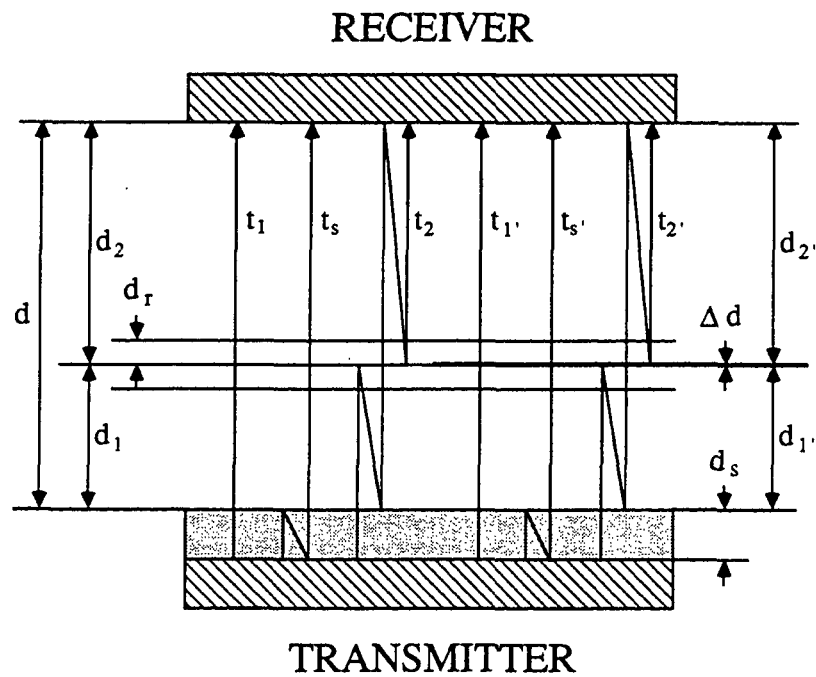


Figure 1.



where  $d_1' + d_2' < d_1 + d_2$

Since sound pulses are traveling in three mediums having different acoustic impedances (water, rubber and air/paper), delayed reflected pulses with respect to the directly transmitted pulse occur. Let's assume that  $d_1$ ,  $d_2$  and rubber thickness  $d_r$  are optimized in such a way that reflected pulses do not overlap in time; traveling times for "reference" pulses (without paper) as depicted on the left side of Figure 1 are described as follows:

$$t_1 = d_s/v_s + (d_1 + d_2 - 2d_r)/v_f + 2d_r/v_r \quad (3)$$

$$t_2 = d_s/v_s + (3d_1 + 3d_2 - 6d_r)/v_f + 6d_r/v_r \quad (4)$$

where  $v_s$ ,  $v_f$  and  $v_r$  are, respectively, the shim, fluid and rubber sound velocities. In a similar manner, traveling times for "paper" pulses as shown on the right side of Figure 1 are

$$t_1' = d_s/v_s + (d_1' + d_2' - 2d_r)/v_f + 2d_r/v_r + \Delta t \quad (5)$$

$$t_2' = d_s/v_s + (3d_1' + 3d_2' - 6d_r)/v_f + 6d_r/v_r + \Delta t \quad (6)$$

in which  $\Delta t$  is the transit time in paper. From equation (3) and (4), we get:

$$\delta t_{21} = t_2 - t_1 = 2(d_1 + d_2 - 2d_r)/v_r + 4d_r/v_r \quad (7)$$

Similarly, from (5) and (6),

$$\delta t_{2'1'} = t_2' - t_1' = 2(d_1' + d_2' - 2d_r)/v_r + 4d_r/v_r \quad (8)$$

Subtracting (8) from (7) and solving for  $\Delta d$ , the caliper is,

$$\Delta d = 0.5 v_f [\delta t_{21} - \delta t_{2'1'}] \quad (9)$$

The transit time in paper is calculated from the subtraction of (3) from (5), i.e.,

$$\Delta t = (t_1' - t_1) + \Delta d/v_f = \delta t_{1'1} + 0.5 [\delta t_{21} - \delta t_{2'1}] \quad (10)$$

Finally, the out-of-plane bulk longitudinal velocity in paper is

$$v_p = \Delta d/\Delta t \quad (11)$$

from which the elastic constant  $C_{33}$  is obtained. Equations (9) and (10) indicate that  $\Delta d$  and  $\Delta t$  can be determined providing that  $v_f$  is a constant of the system at constant temperature. In practice, time measurements depend upon mechanical pressure effects at paper-rubber interfaces. Hence, the fluid velocity must be reformulated as an effective fluid velocity  $kv_f$  in which  $k$  is a calibration factor. At constant temperature, the effective velocity is found by using the following equation:

$$kv_f = (2\Delta d_{sp}) / [\delta t_{21} - \delta t_{2'1}] \quad (12)$$

where  $\Delta d_{sp}$  is the soft-platen caliper (assumed to be known). If measurements are collected from a large number of samples having different thicknesses, an average effective velocity is obtained.

#### Principles: Temperature Dependent Measurements

Assuming that temperature variations in both wheels are nearly identical, temperature variations can be monitored in one wheel only. Since sound velocity in any material strongly depends upon temperature, a plastic delay line or shim conveniently located in front of the transmitter can be used to monitor temperature. As seen in Figure 1, the time delay between the directly transmitted pulse ( $t_1$ ) and the first internally reflected pulse in the shim ( $t_s$ ) depends solely on temperature variations in it. Thus, whatever paper specimen is or is not in the nip, the shim time delay at temperature  $T$  is

$$\delta t_{1s}(T) = t_s(T) - t_1(T) = t_s'(T) - t_1'(T) \quad (13)$$

Providing the calibration curves  $t_1(T) = f(\delta t_{1s}(T))$  and  $\delta t_{21}(T) = f(\delta t_{1s}(T))$  determined over the full temperature operating range, equations (9) and (10) can be reformulated to take into account temperature effects:

$$\Delta d(T) = 0.5 k_{vf}(\delta t_{1s}(T = T_R)) \delta t_{21}(\delta t_{1s}(T = T_R)) [1 - \delta t_{2'1}(T)/\delta t_{21}(\delta t_{1s}(T))] \quad (14)$$

$$\Delta t(T) = [t_{1'}(T) - t_1(\delta t_{1s}(T))] + 0.5 [\delta t_{21}(\delta t_{1s}(T)) - \delta t_{2'1}(T)] \quad (15)$$

where  $T_R$  is a reference temperature (e.g., room temperature). It is interesting to note that explicit temperature measurements are not required in the ultrasonic temperature compensation technique.

### Measurements and Results

Two commercially available unfocused immersion piezoelectric transducers were used to launch and receive pulses. Peaking at 1 MHz, they were embedded in molded urethane (hard rubber) tires filled with water. A wave function generator was used to launch pulses (1 MHz, one full-cycle sinusoidal pulses). Incident pulses were amplified with a wide-band power amplifier. Received waveforms were preamplified and captured on a one-to-one basis with a GPIB controlled 2432 Tektronix scope. The scope time resolution was set to 50 ns and its vertical resolution was 8 bits. Signal averaging (32 successive recordings) was used to improve the signal-to-noise ratio. Averaged waveforms were transferred to a 386 computer via the GPIB interface. Data acquisition and waveform analysis were handled with a data acquisition and analysis software package (W.A.V.E.). Hardware improvements in the two-transducer arrangement have resulted in an undispersed reflected pulse ( $t_2'$ ) when compared to the directly transmitted pulse ( $t_1'$ ). This allowed cross-correlation measurements using full-length waveforms in order to achieve very accurate time delay determination ( $\pm 1$  ns).

In order to demonstrate the measurement capabilities of the fluid-filled wheels technique, data were gathered for 29 different paper samples from 50 to 1800  $\mu\text{m}$  thickness. Samples were divided into four categories: fine papers, linerboards, mediums and heavy weight boards. Since all measurements were done at room temperature, equations (9) and (10) were used to evaluate caliper and transit time.

Figure 2 shows the fluid-filled wheels (FFW) and hard-platen (HP) calipers as a function of the soft-platen (SP) caliper. Agreement is better between FFW and SP calipers than it is between HP and SP calipers. Moreover, very good agreement is achieved over the full thickness range. This later observation is confirmed by the graph depicting the FFW

caliper offset (%) vs. SP caliper (Figure 3). Figure 4 represents the FFW caliper as a function of the basis weight. As the relationship is linear, this suggests that FFW caliper measurements are pressure insensitive. Next, the FFW caliper was plotted against the transmitted pulse ( $t_1$ ) center frequency (Figure 5). This graph indicates that the center frequency is inversely related to caliper or traveling distance in the specimen. As seen in the next section, sound attenuation in materials is directly related to frequency, and as such, Figure 5 results confirm that sound attenuation in paper is inversely related to the caliper.

Using FFW caliper and transit time measurements, the FFW velocity was computed. It is reported in Figure 6 as a function of the soft-platen velocity (IPST out-of-plane longitudinal velocity tester). The FFW velocity offset (%) vs. SP velocity is shown in Figure 7. Agreement is generally good for all papers with the exception of fine papers. No reliable explanation has been found so far to explain the discrepancy for fine papers. However, since FFW measurements are more consistent than soft-platen measurements (no calibration with thin metal shim is required in the FFW instrument), it is believed that the FFW velocity is a more reliable quantity.

In the March 1990 report, the validity of cross-correlating two pulses having different center frequencies was discussed. This problem is unavoidable because transit time measurements are absolute. A fully functional technique has been developed to overcome this problem by evaluating the transit time from the time delay between pulse leading edges. By doing so, good agreement is achieved for fine paper velocities, but not for heavy weight ones. This has raised questions on the exactness of absolute velocity measurements. Contrary to caliper data which can easily be calibrated, velocity measurements can hardly be calibrated. One possible way to achieve this goal would be to verify the FFW elastic modulus (density times velocity squared) against the mechanical elastic modulus obtained from ZD tensile measurements.

The fluid-filled wheels experimental setup will be the subject of a presentation at the Ultrasonics International '91 Conference and Exhibition, Le Touquet, France, July 1-4, 1991.

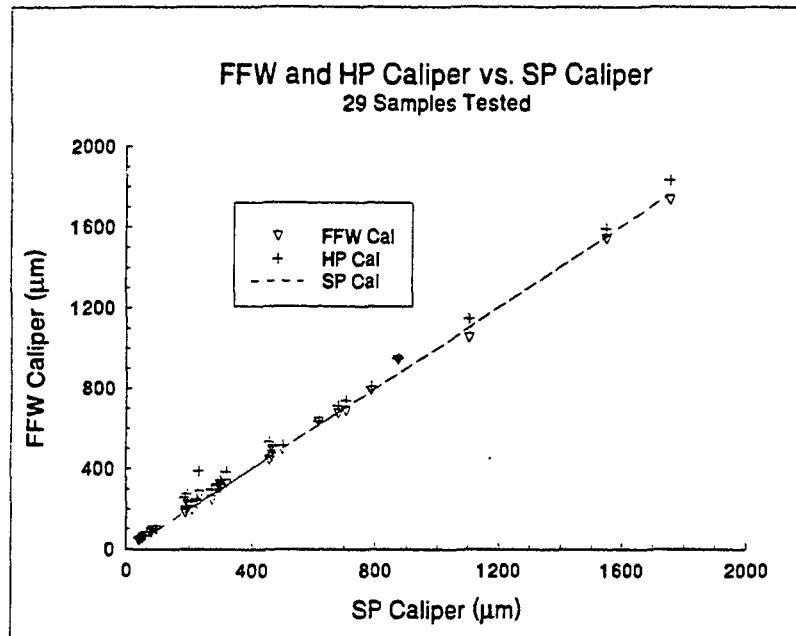


Figure 2.

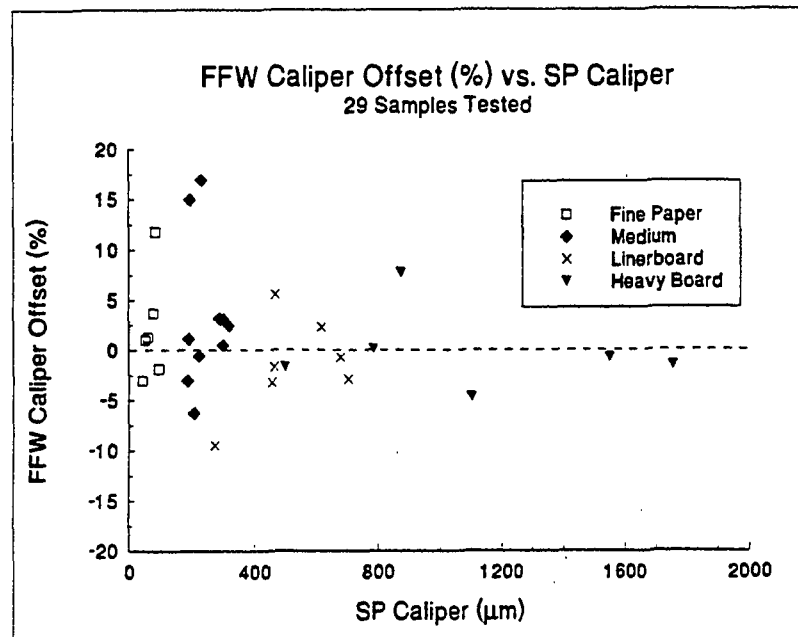


Figure 3.

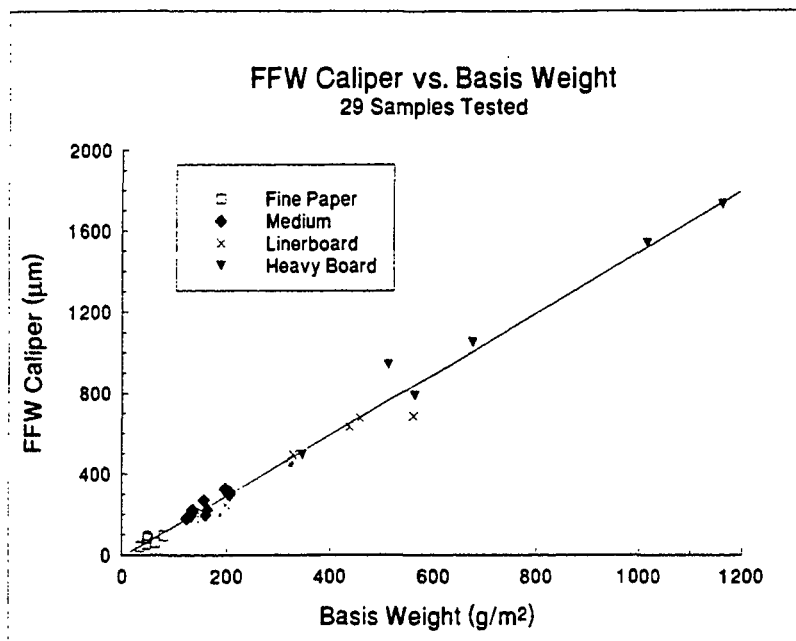


Figure 4.

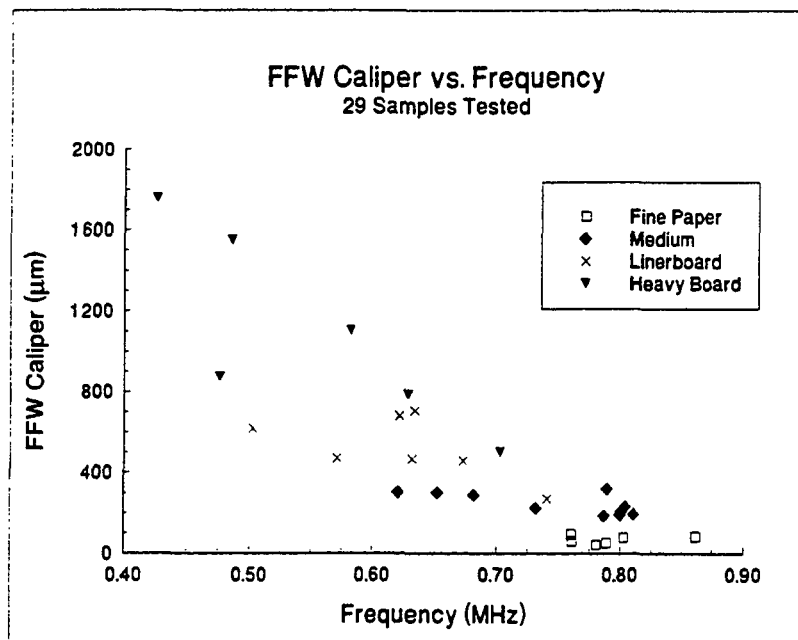


Figure 5.

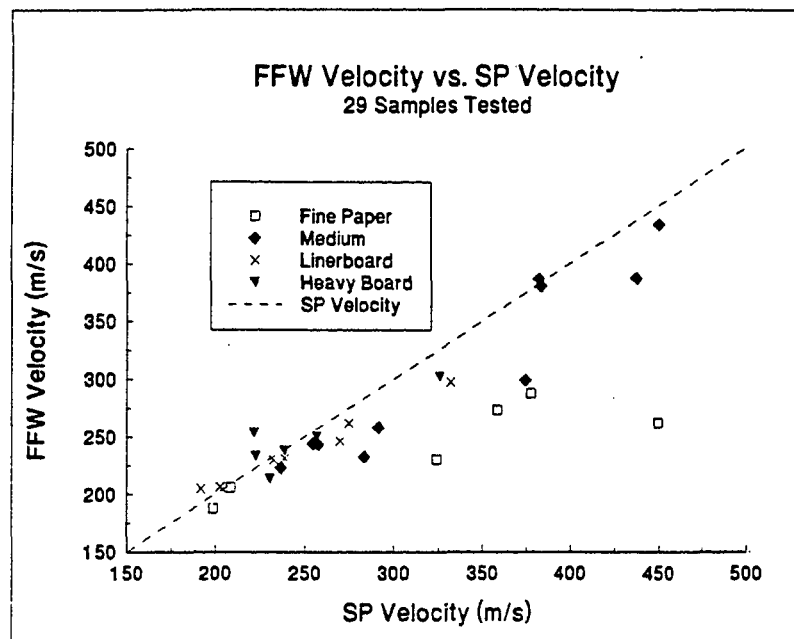


Figure 6.

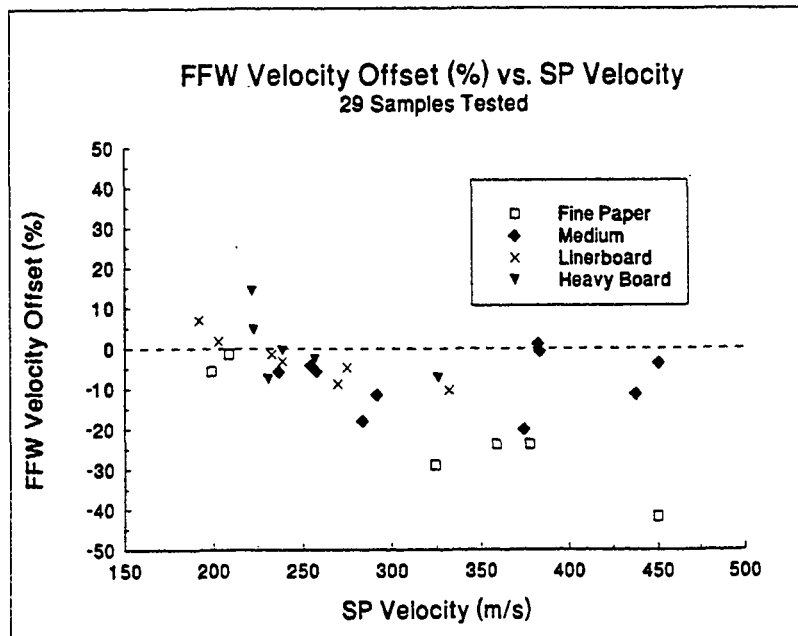


Figure 7.

b. Out-of-plane Sound Dispersion and Attenuation in Paper

Frequency domain analysis of the directly transmitted pulses recorded with the fluid-filled wheels setup has generated an interest in the desire to understand fundamentals of sound dispersion and attenuation in paper materials. Since sound dispersion and attenuation in materials can be predicted from either quantity using the so-called Kramers-Kronig relationships, preliminary results reported here indicate that sound reflection can be indirectly evaluated from dispersion and attenuation measurements.

Frequency Domain Analysis

Consider the time domain waveforms illustrated in Figure 8. The reference pulse corresponds to the directly transmitted pulse recorded with no paper in the nip ( $t_1$ ). For demonstration purposes, the sample pulse corresponds to the directly transmitted pulse ( $t_1$ ) recorded with a 69 lb linerboard specimen. Using a fast Fourier transform algorithm (FFT), frequency domain analysis of the waveforms can be achieved. Amplitude ( $U$ ) spectra and phase ( $f$ ) spectra as a function of frequency for reference and sample waveforms are displayed in Figure 9 and 10, respectively. From the amplitude spectra, one can see a large change in amplitude due to sound attenuation. Moreover, Figure 9 clearly shows the shift toward a lower frequency for the sample center frequency. This is also a manifestation of sound attenuation. An analysis of the phase spectra (Figure 10) indicates that the sample phase spectrum is approximately linearly related to frequency. This means that sound dispersion in the linerboard specimen is small, at least up to 1.5 MHz. Since no experimental results are available above 1.5 MHz, one cannot postulate for the sound dispersion at higher frequencies. The larger negative slope for the sample spectrum is in agreement with the extra traveling time in the sample.

Principles of Sound Dispersion and Attenuation

Sound dispersion in materials refers to the phase velocity variation as a function of frequency. In agreement with the FFW setup, the phase velocity can be described as:

$$v_p(f) = \omega/\beta = v_f/[1 + (v_f\Delta\phi(f))/(2\pi f \Delta d)] \quad (16)$$

where  $\Delta\phi(f) = \phi_r(f) - \phi_s(f)$ ,  $\omega = 2\pi f$ ,  $\beta$  is the propagation constant of the specimen and



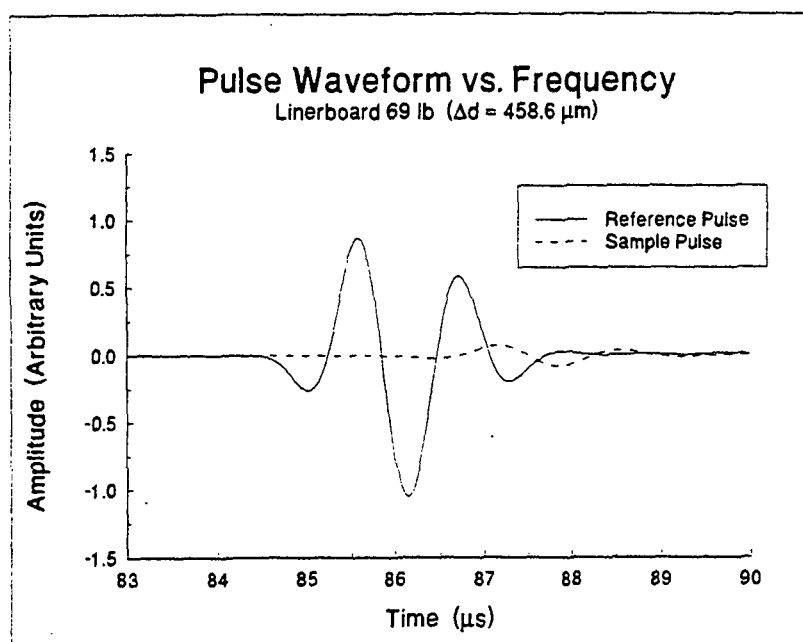


Figure 8.

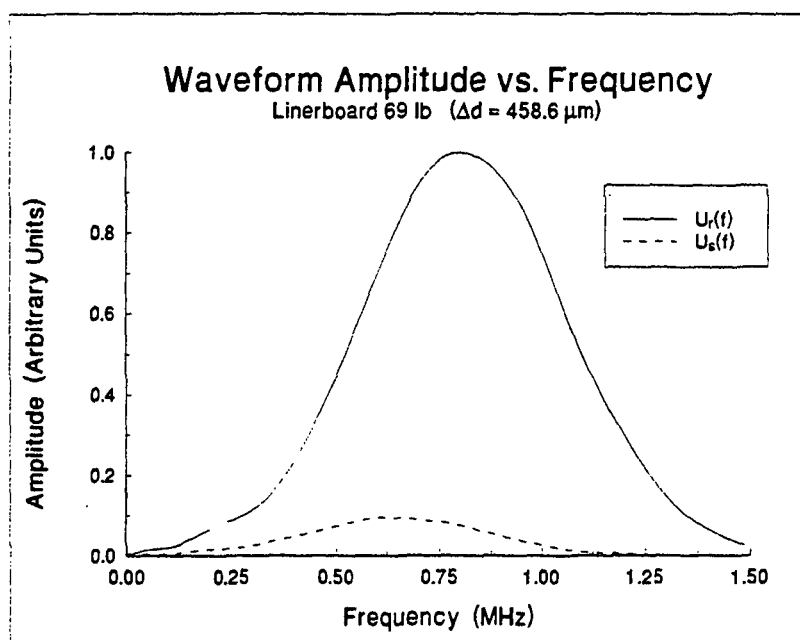


Figure 9.

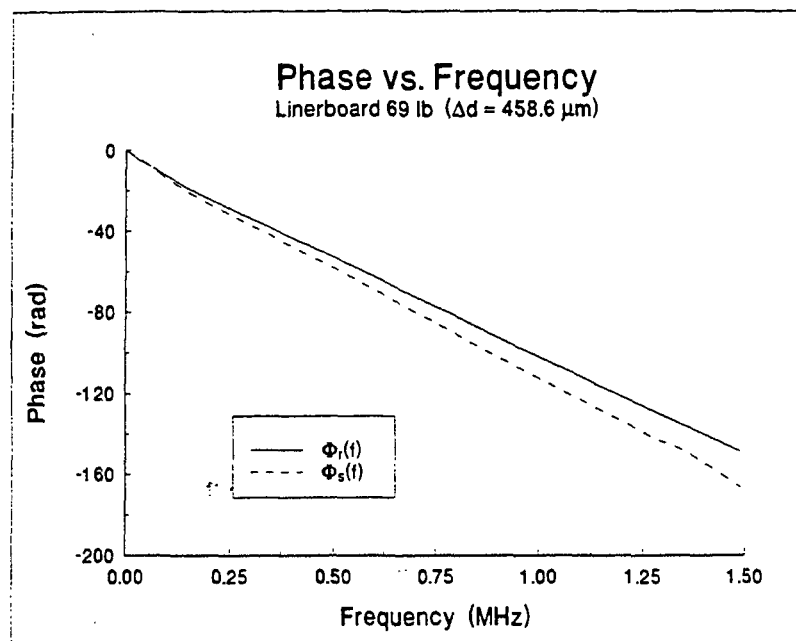


Figure 10.

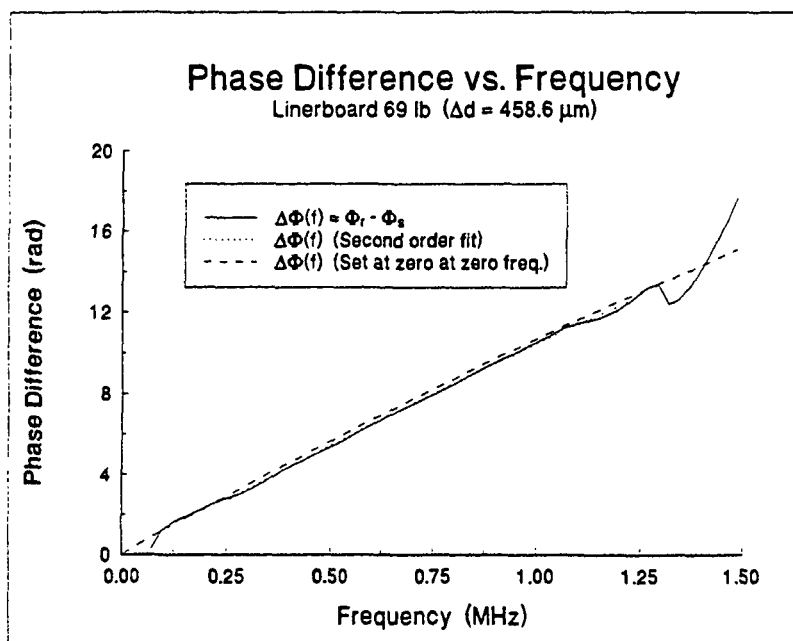


Figure 11.

$v_f$  is the fluid velocity (velocity of the reference medium when no sample is in the nip). One can also define the group velocity which corresponds to the rate of change of the phase velocity as the sound waves propagate through the specimen. The group velocity is:

$$v_g(f) = d\omega/d\beta = v_f / [1 + (v_f/(2\pi \Delta d))(d(\Delta\phi(f))/df)] \quad (17)$$

For a non-dispersive material  $v_g(f)$  is equal to  $v_p(f)$ . Sound velocity in paper as defined by equation (11) is a frequency independent approximation to the phase velocity. In other words, equation (11) is valid if dispersion is very small.

Since materials in which ultrasonic waves propagate are more or less lossy, the amplitude of these waves decreased with distance. This phenomenon corresponds to sound attenuation. For a material with thickness  $Dd$  and sound reflection coefficient  $R$ , the sound attenuation is given by

$$\alpha(f) = (-1/\Delta d) \ln[ (U_s(f)/U_r(f))/(1 - R^2) ] \quad (18)$$

For an unbounded material, sound attenuation is the sufficient and necessary condition to develop dispersion. Fortunately, relationships are available to predict attenuation from dispersion information and vice-versa (Kramers-Kronig relationships). Using these relationships, attenuation as predicted from dispersion is,

$$\alpha(f) = ((-\pi f^2)/(2\Delta d)) (d(\Delta\phi(f)/f)/df) \quad (19)$$

Equating (18) and (19) and solving for  $R$  provides an indirect way to evaluate the reflection coefficient from dispersion and attenuation measurements.

### Sound Dispersion Measurements

Using the phase spectra shown in Figure 10, the phase difference  $\Delta\phi(f)$  has been calculated. The result is shown in Figure 11 (solid line). Neglecting end effects at low and high frequencies (due to inaccuracy of measurements in these regions) one can see that the phase difference is approximately linearly related to the frequency. This suggests that there is very little dispersion in this frequency range. A second order polynomial fit has been performed between 0.25 and 1.25 MHz (dotted line). Since the phase difference must

be zero at zero frequency, the fitted curve intercept has been reset (dashed line). Using equations (16) and (17), the phase and group velocities can be computed and the results are depicted in Figure 12. As it can be seen, dispersion is small. As a means of comparison, the sound velocity in paper ( $v_p$ , equation 11) has been included in the graph. It is seen that this approximated result (frequency independent) is justified.

The attenuation coefficient has been calculated using equations (18) (calculated from Figure 9 amplitude spectra) and (19) (deduced from Figure 11 phase difference spectrum). Results are shown in Figure 13. The solid line corresponds to the indirect measurement. Direct measurements for  $R = 0$  and  $R \neq 0$  reflection coefficient correspond to the long and short dashed lines, respectively. Neglecting end effects once again, agreement between directly and indirectly calculated attenuation coefficients is very good. The reflection coefficient spectrum used in the calculations is shown in Figure 14. In the region of interest, this coefficient is nearly frequency independent. Its average value is around 0.875. This means that 87.5 % of the incident wave is reflected. It is assumed in this analysis that the reflection coefficient is an average coefficient of both reflective surfaces since there is no way to discriminate their respective coefficients.

In order to verify the validity of the predicted reflection coefficient, a pulse-echo experiment has been devised to gather direct measurements of the reflected signal at the rubber-paper interface. A pulser-receiver is used. Unfortunately, acoustic impedances of hard-rubber and paper are very different; therefore, direct reflection analysis has failed to detect any reflection dependency on paper. In other words, the measured reflection coefficient is independent of the paper surface. Measurements have not yet been collected with soft-rubber tires.

Work is in progress to analyze sound dispersion and attenuation measurements collected from the 29 available paper specimens. Results on sound dispersion and attenuation in paper materials will be reported at the 1991 International Paper Physics Conference, Hawaii, September 22-27.

c. Temperature and Moisture Effects on ZD elastic Properties.

One problem that needs to be addressed for on-line out-of-plane elastic property measurements is the combined effect of temperature and moisture on ultrasonic

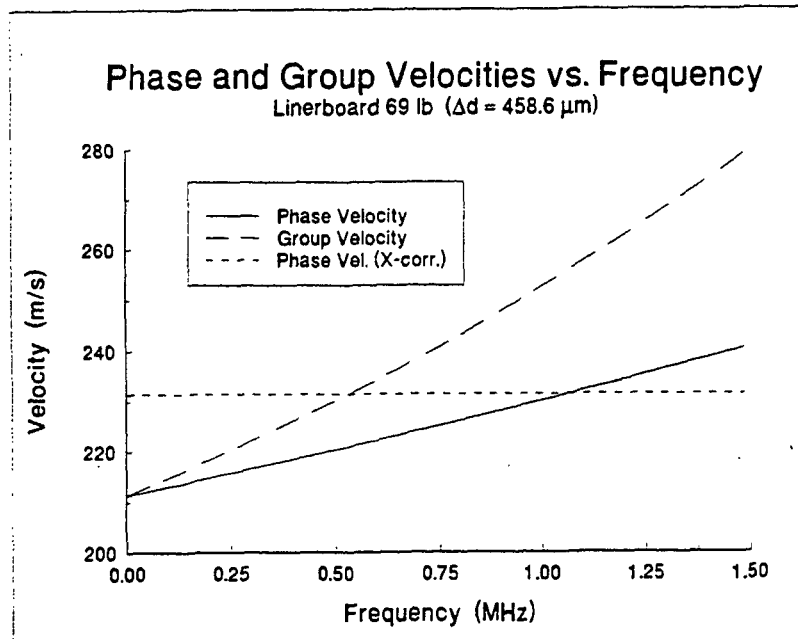


Figure 12.

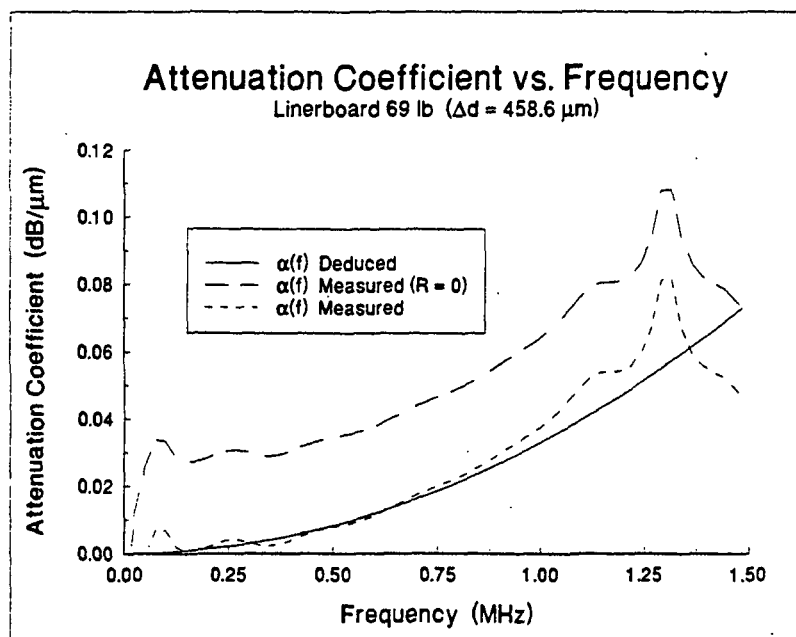


Figure 13.

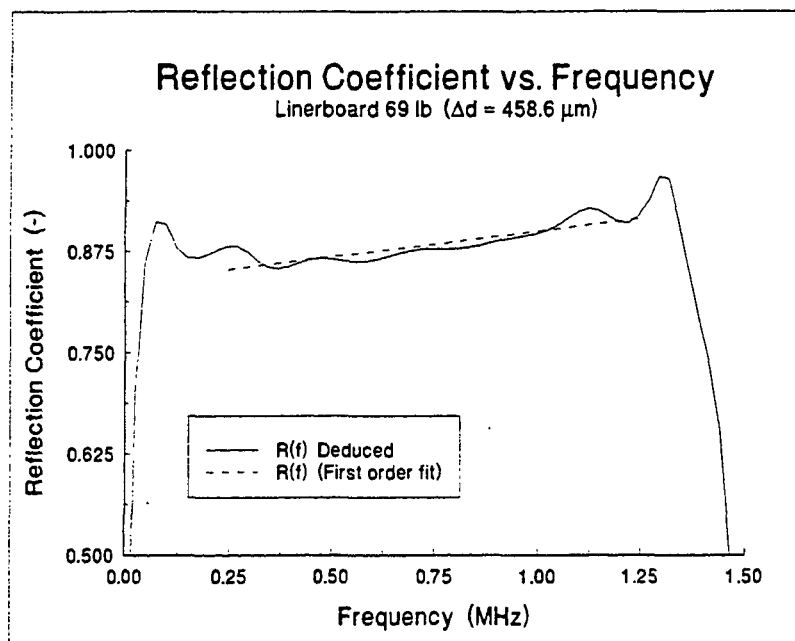


Figure 14.

measurements. In order to address the problem on a systematic basis, an experiment has been designed to collect FFW caliper and velocity in a controlled temperature/humidity chamber. A duplicate of the FFW setup has been built for that purpose. A remote-controlled arm will allow 90-degree rotation of the wheels to allow out-of-the-nip sample conditioning in the chamber.

## 2. Non-destructive Microwave Evaluation of Paper Materials

### a. Microwave Determination of Fiber Orientation

The main interest in this project lies in the development of a non-contact sensing technique to measure fiber orientation independent of density and moisture content variations. This a difficult task to achieve. A thorough analysis of the various methods currently available or under development for fiber orientation measurement shows that high frequency microwave (around 100 GHz) is the most serious contender. While in-plane ultrasonic characterization provides maximum stiffness lean angle, measurement dependency upon induced drying stresses prohibits direct relationship with fiber orientation. X-rays are sensitive to cellulose crystal orientation and thus provide indirect fiber orientation. Visible range methods are inadequate for thick papers. The far-infrared method is moisture content dependent and unsuitable for basis weight exceeding 200 g/cm<sup>2</sup>. Low-frequency microwave cavity measurements should provide true fiber orientation but would not have the potential for on-line use.

Microwave measurements are basis weight and moisture content dependent. Three free-space techniques have been proposed to overcome these drawbacks. The first two imply high frequency transmission and reflection measurements as a function of angle with respect to machine direction (see Figure 15). In the third method, it is proposed to evaluate scattering at blazed angle with respect to the paper plane; maximum scattering occurs at 90 degrees off expected fiber orientation. In the later case, periodicity of the structures in paper would be investigated.

In that regard, an exploratory research proposal is being prepared by GTRI. With the exception of a special purpose sample holder, all equipment required for the demonstration experiments is available. Newsprint, linerboard and thickboard samples will be tested. Results will be compared to in-plane ultrasonic measurements collected at the Institute.

## GTRI Freespace Reflection and Transmission Variable Range Arch : Bistatic

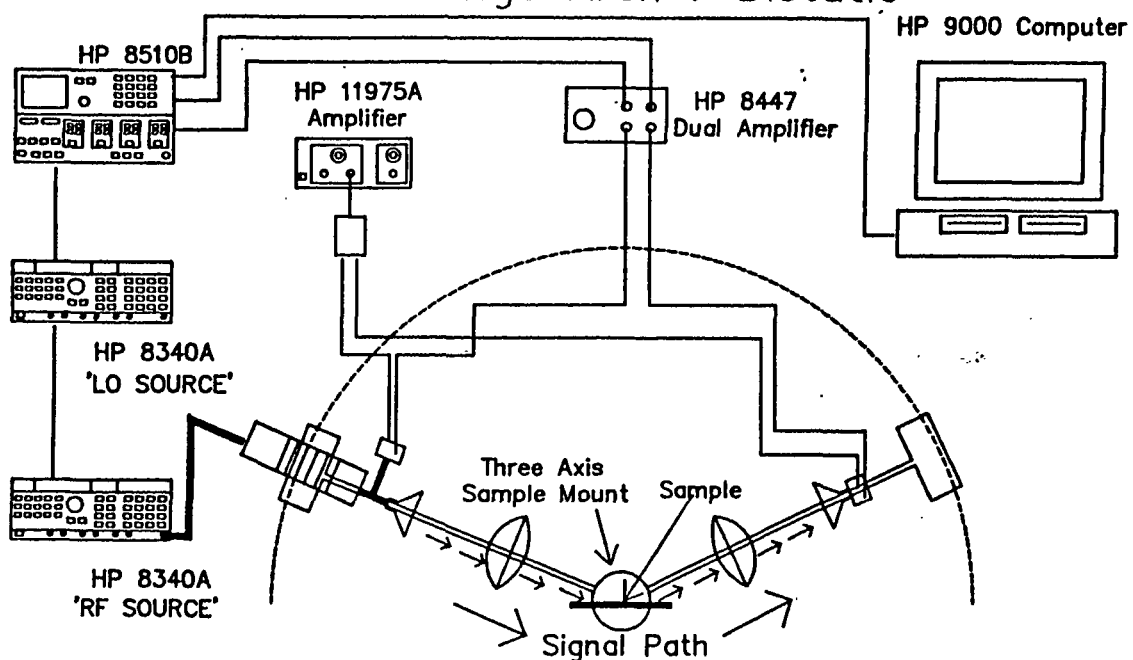


Figure 15.



b. Joint Research Project IPST-GTRI

The Institute of Paper Science and Technology has limited capabilities and expertise in microwave characterization of materials. Hence, a joint research program has been established with the Georgia Tech Research Institute (GTRI) of the Georgia Institute of Technology. The GTRI has two independent laboratories dealing with microwave-related activities (Microwave and Antenna Laboratory and Electromagnetic Effectiveness Laboratory). It has been agreed that both labs would actually be involved in the joint research project. They are represented by Dr. R. Moore, Senior Research Scientist (EEL), and G. Hopkins, Junior Research Engineer (MAL). Dr. Moore has a long-standing expertise in microwave characterization of materials.

3. Plasma Processing of Paper Materials

Exploratory Research Proposal on Investigation of Paper Internal Properties using Microwave Plasma-assisted Etching (P. Brodeur and J. Waterhouse)

As part of the development of a new research area at the Institute, namely plasma processing of paper materials, an exploratory research project has been devised to investigate paper internal properties. Since these properties can hardly be measured by non-invasive techniques, surface removal methods, although destructive, are appropriate.

Surface grinding has been tested in the past. However, a certain level of uncertainty remains due to possible alteration of some of the properties to be evaluated. It is more likely that some of the ground material is not thrown away in the process, but rather captured by the bulk material, thus altering its porous nature. Surface grinding is a directional process which may not be particularly suitable for an anisotropic material like paper. Also, induced stresses and heating effects cannot be disregarded.

In view of the concerns associated with the mechanical aspect of surface grinding, alternative and more gentle surface removal techniques need to be explored. Many non-mechanical layer removal techniques have been developed for the semiconductor industry, the most prominent of which are based on plasma-assisted etching, laser ablation and ion milling. A preliminary investigation of these techniques has shown that plasma-assisted etching is the most promising technique for removing layers of paper materials while

evaluating internal properties.

A plasma is a combination of neutral and positively and negatively charged particles; it is electrically neutral on a macroscopic scale. The combination of neutral species adsorption and positive ion bombardment results in surface chemical reaction. If the products of the surface reaction are volatile, they leave the surface and etching occurs. Plasma-assisted etching was recently tested on paper materials by Sapieha et al. (1988)\*, who performed etching experiments using a "large volume microwave plasma" system. Uniformity of the treatment is insured by rotating the paper specimen. Since the major task of Sapieha's work was to determine the best plasma parameters to etch paper, his results are mostly qualitative. Hence, much remains to be done in order to assess the technique in a quantitative manner. Since paper is porous, one of the problems that needs further examination is the extent of damage beyond the etched surface. In other words, what is the level of uniformity that can be achieved with this technique? Are fine particles etched before fibers? What are the implications of the unavoidable zero-percent moisture content during processing (vacuum effect)?

The research project has two main objectives:

- 1) Evaluate the microwave plasma-assisted technique as a new means to study internal properties of paper.
- 2) Determine internal stress variation in the thickness variation as obtained from plasma-assisted etching and surface etching experiments.

It is planned to investigate three linerboard materials (33, 42 and 69 lbs). Up to 50 % of the material will be removed from each specimen (10, 20, 30, 40 and 50%). From curvature measurements, thickness direction residual stress distribution is obtained, from which thickness direction internal stress distribution is calculated. Using ultrasonic techniques, out-of-plane and in-plane elastic measurements will be gathered as well. Plasma etching experiments will be conducted at Ecole Polytechnique de Montreal.

\* Sapieha, S., Wrobel, A.M. and Wertheimer, M.R., Plasma-assisted Etching of Paper, Plasma Chem. Plasma Process. 8 (1988) 331-346.

#### 4. Acoustic Radiation Pressure Effects on Fluid Suspended Fibers

##### a. Motion of Fluid Suspended Fibers in a Standing Wave Field

A paper titled "Motion of Fluid Suspended Fibers in a Standing Wave Field" has been completed and submitted for publication. The paper should appear in the May 1991 issue of Ultrasonics. It presents a model aimed at explaining and predicting some experimental results obtained with water-suspended cellulosic fibers subjected to an ultrasonic standing wave field. Under this condition, it is well known that fibers migrate to preferred sites at stable equilibrium positions and reorient to stable equilibrium angular positions. The model shows that acoustic layering and reorientation are dominant effects for long and short fibers, respectively. Since fiber length and width are related for wood fibers, it was not possible to test the model against these two quantities. This work supports the experimental development of acoustic fiber alignment techniques.

##### b. Acoustic Fiber Alignment

As previously introduced, the successful development of an acoustic fiber-alignment technique would play a major role in the development of a new fiber analyzer system. In the preliminary phase, it is planned to investigate acoustic alignment of slow moving fibers diluted in distilled degassed water at room temperature. Figure 16 shows a conceptual view of the proposed arrangement. Fibers are moving in a small 10 X 10 mm<sup>2</sup> square cross-section transparent tube. Two resonators are necessary to achieve three-dimensional alignment. Both have 10 X 100 mm<sup>2</sup> cross-sections. Sound attenuation should not be a problem at low consistency. Hence, standing waves in each resonator can be produced with a transducer-reflector assembly. Assuming a flow speed of 10 mm/s, the dwelling time in each resonator is 10 s.

Since acoustic layering and reorientation occur simultaneously, several parameters must be optimized to promote fiber alignment. They are: resonator length, flow rate, acoustic wavelength and acoustic power. Three acoustic wavelengths will be tested ( $\lambda = 20\text{mm}$ , 5mm and 2.5 mm). Assuming an approximate sound velocity of 1500 m/s in water at room temperature, this can be achieved with transducers resonating at 75 kHz, 150 kHz and 300 kHz, respectively. The Institute does not have full capabilities in transducer design, and as

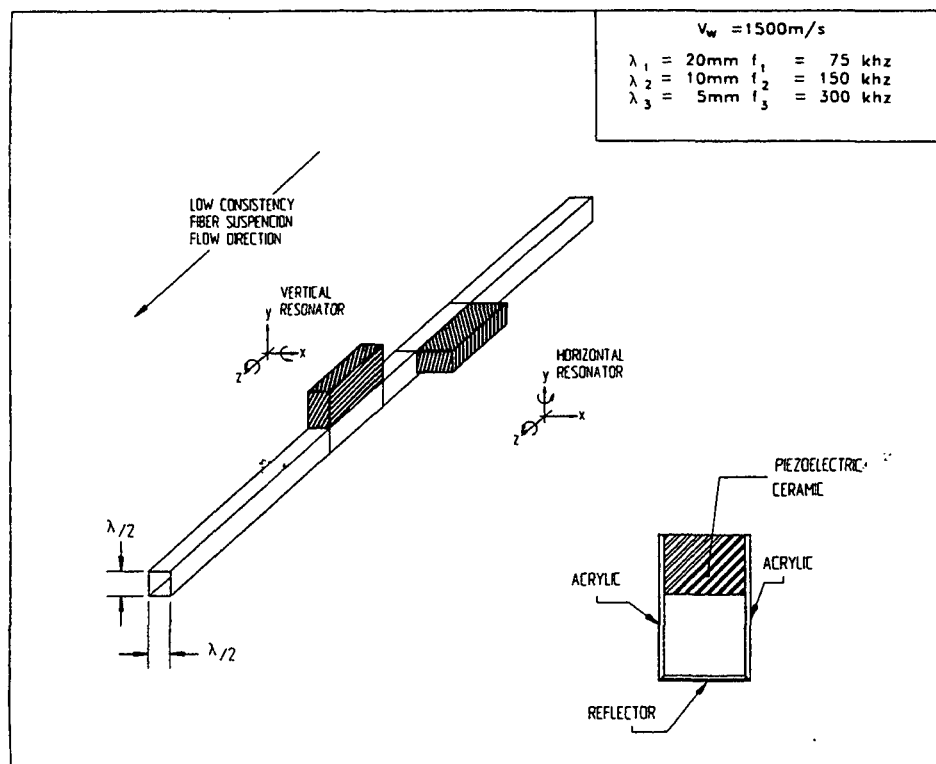


Figure 16.

such, a small consulting firm, Sonic Concepts, has agreed to design and build the piezoelectric ceramic transducers. Resonators and flow loop system will be developed at the Institute. A laser-based optical system will be used to monitor visible light polarization effects during the reorientation process. Synthetic fibers with fixed diameter and variable length, and variable diameter and fixed length, will be tested first to fully understand fiber alignment dependency on dimensions.

STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

STATUS REPORT

FOR

PROJECT 3469

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

April 4, 1991

## PROJECT SUMMARY

Project: STRENGTH IMPROVEMENT AND FAILURE MECHANISMS  
Project Code: STRNG  
Project Number: 3469  
Division: Engineering and Paper Materials Division  
FY 90-91 Budget: \$20,000  
Project Staff: J. Waterhouse, D. Brennan, B. Okoth, S. Clark

### PROJECT OBJECTIVE:

To improve strength properties through a better understanding of the interactions between certain raw material, papermaking, and converting process variables. Specific areas of focus to include formation and internal stresses.

### PROGRAM AREAS:

End Use Performance. Reduced Operating Costs

### RATIONALE:

**FORMATION.** Small scale variations in a number of structural, mechanical, and physical properties are considered to play a vital role in the converting and end use properties of paper and board. To date, significant effort has been devoted to the assessment and measurement of the visual uniformity of paper, but not much is known about the relationship of formation to other converting and end use properties of paper and board. An improvement in our understanding of these relationships would have broad consequences affecting not only improved converting and product performance, but improved paper machine operation and reduced energy consumption.

**INTERNAL STRESSES.** Dimensional stability and curl play a vital role in the converting and end use performance of paper and board. In the

past curl has been treated, somewhat superficially, as a felt-wire side problem. With the recent advances we have made in ultrasonic non-destructive testing (particularly polar diagrams) and the measurement of internal and residual stresses, we are well positioned to obtain a more detailed understanding of how variations in the thickness direction treated as a continuum affect dimensional stability and curl behavior. A better understanding of these factors controlling the dimensional stability and curl behavior of paper and board should lead to improved runnability in converting processes, improved product performance and a reduction in off specification paper and board. Many areas of paper machine performance should also be improved.

#### FY 90-91 GOALS:

##### FORMATION:

- (1) Determine the effect of formation on fracture toughness measurements.
- (2) Write a woodgrain report on the IPST Formation Tester

##### COMBINED STRESSES:

- (1) Complete failure envelope measurements at different levels of refining wet pressing.
- (2) Write a wood grain report on a new device for combined out-of-plane stress measurements.

##### COMPRESSIVE STRENGTH IMPROVEMENT

- (1) Write a wood grain report detailing recent results on strategies for compressive strength improvement.



## INTERNAL STRESSES

- (1) Determine the suitability of excimer lasers and plasma etching as layer removal techniques.
- (2) Write a wood grain report on internal and residual stresses.

## RESULTS LAST PERIOD: (April 1990 - November 1990)

### FORMATION:

1. Software Developments:
  - a) Calculation of coefficient of variation of optical and mass density for different size apertures.
  - b) Established interface with Tracor Northern Image Analyzer.
  - c) Established interface with Axum - graphics package.
  - d) Developed reel strip scanning and analysis.
  - e) Motion increased for both continuous and discrete modes.
  - f) Formation measurements made on fracture toughness and tensile samples.
2. Hardware Developments:
  - a) Reel to Reel system designed and fabricated for measuring formation on reel strip samples.
3. Other

- a) Miami University Pulp and Paper Foundation "Formation and End Use Performance" June 1990.
- b) API Publications: Measurement Technology Program Report No. 70 Parts II & III, July 1990

#### COMBINED STRESSES:

- 1. Failure envelope measurements have been completed for MD samples at different levels of refining and wet pressing.

#### COMPRESSIVE STRENGTH IMPROVEMENT

- 1. A draft wood grain report has been completed surveying recent results on strategies for compressive strength improvement.
- 2. "Effect of Synthetic Fiber and Binder Addition on the Strength Losses Associated with Corrugating Medium" Materials Research Society - Symposium Proceedings Volume 197 1990.

#### INTERNAL STRESSES

- 1. Joint research proposal developed with Dr. Pierre Brodeur to determine the suitability of plasma etching as layer removal technique.
- 2. "The Effect of Internal Stresses on the Modification of Paper Properties by Calendering" Renee Ebert - Student related Research.
- 3. "Internal Investigation of Elastic Properties of Paper Using Non-Mechanical Layer Removal Techniques" Student A390 Problem Todd Schwantes.

## GOALS FOR THIS PERIOD: (November 1990 - April 1991)

## FORMATION:

- (1) Determine the effect of formation on fracture toughness measurements.
- (2) Write a wood grain report on the IPST Formation Tester

## COMBINED STRESSES:

- (1) Write a wood grain report on a new device for combined out-of-plane stress measurements.

## INTERNAL STRESSES:

- (1) Determine the suitability of plasma etching as a layer removal technique.
- (2) Write a wood grain report on internal and residual stresses.

## RESULTS THIS PERIOD:

## FORMATION:

- (1) A draft wood grain report on the IPST Formation Tester has been completed.
- (2) An approximate method to predict the variability of mass density from the variability of optical density has been developed.

**INTERNAL STRESSES:**

- (1) A joint proposal has been developed with Dr Pierre Brodeur and submitted to Ecole Polytechnique to determine the suitability of plasma etching as a layer removal technique. It is proposed to compare this technique with surface grinding which has been previously used as a layer removal technique.

**GOALS FOR NEXT PERIOD: (April 1991 - November 1991)**

Project 3469 will be closed as of June 30, 1991, and the remaining wood grain reports will be completed by that date.

**COMBINED STRESSES:**

- (1) Write a wood grain report on a new device for combined out-of-plane stress measurements.

**INTERNAL STRESSES:**

- (1) Write a wood grain report on internal and residual stresses.

**OTHER PROJECTS & ACTIVITIES:**

1. API Contract work for evaluation of on-line formation testers.
2. NSF Research Grant "Role of Melamine-Formaldehyde in Currency Papers" A presentation of the goals, the research to be carried out, and achievements to date, is to be given at the National Science Foundation, Washington D.C. on March 16, 1991.
3. A joint paper with T. Barrett, University of Iowa "Aging Characteristics of European Handmade Papers" has been submitted to TAPPI Journal.

4. Prepared a joint paper with T. Bither, Ph.D student "A Device for Continuous Sheet Restraint During Pressing and Drying" for presentation at Progress in Paper Physics Seminar, Kalamazoo, Sept. 1990 and submission to a refereed journal.
5. The following two papers have been submitted and accepted for presentation at the 1991 International Paper Physics Conference in Hawaii. "Strength Development by Refining and Wet Pressing" co-authored with Thomas Bither and "The Failure Envelope of Paper when Subjected to Combined Out- Plane-Stresses"

## STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

### Project 3469

#### INTRODUCTION

This project is comprised of four distinct but interconnected project areas namely: Compressive Strength Improvement, Formation, Combined Stresses, and Internal Stresses in Paper and Board. Following PAC Committee interests and recommendations our work has more recently been focussed in the areas of Formation and Combined Stresses.

During the last year we have devoted our effort to improving formation measurements, investigating the effects of refining and wet pressing on combined out-of-plane stress measurements, and preparing wood grain progress reports on "Strategies for Improving Compressive Strength" and "Formation Measurements"

#### COMPRESSIVE STRENGTH IMPROVEMENT

A paper "Effect of Synthetic Fiber and Binder Addition on the Strength Losses Associated with Corrugating Medium" has been published as Technical Paper Series #356, and also in Materials Research Society Symposium Proceedings Volume 197 "Material Interactions Relevant to the Pulp, Paper, and Wood Industries" edited by D.F. Caulfield, J.D. Passaretti, and S.F. Sobczynski.

A woodgrain progress report has been prepared on "Strategies for Improving Compressive Strength" and is awaiting publication. The summary of this progress report is included at the end of this report.

#### FORMATION

A draft woodgrain progress report has been prepared on our work to date in the area of formation, which has been mainly devoted to the development of the IPST Formation Tester a device for measuring the optical and mass density variability in paper.

During the last year a number of software and hardware improvements have been made to the IPST Formation Tester. The software improvements include:

- A. The calculation of coefficient of mass and optical density for different aperture sizes.
- B. The establishment of an interface with the Tracor Northern Image Analyzer, and Axum a graphics package.
- C. Developed reel strip scanning and analysis.

The hardware improvements included a reel-to-reel system designed and fabricated for measuring formation on reel strip samples.

Our beta source promethium 147 is now approaching its half life of 2.4 years. Efforts to procure a new beta source having an improved performance from Amersham International were, unfortunately, not successful. The new source which Amersham International recommended, i.e. a 1mm diameter 3-4 mCi source, was supposed to have one and a half times the activity of our present source. When this source was installed it was found that its performance was 15% lower than our old source! We have decided, pending negotiations with Amersham International, that we will re-order the same type of source we had previously, i.e. 50 mCi on a 5mm diameter foil with a 2  $\mu$ m silver window.

#### Optical and Mass Density Measurements

The area of formation measurement is very controversial, in part because there is no universally accepted way of measuring formation or defining an index of formation. We have suggested, as have others, e.g. see Dodson C. T. (1), and Dodson C. T. and Fekih, K. (2) that a suitable index of formation is simply the coefficient of variation or its inverse. This can be used for both optical and mass density measurements. It has been shown by Herdman, P. T. (3) and more recently by Dodson, C. T. (1) that for an ideal network of random fibers that the coefficient of variation of mass density is dependent on fiber coarseness, a fiber length dependent parameter, and inversely proportional to the square root of grammage. The latter relationship, i.e. that  $C.V.(W)$  is proportional to the inverse

square root of grammage  $1/\sqrt{W}$  is shown in Figure 1, surprisingly, it does not appear that this relationship has yet been verified by other researchers.

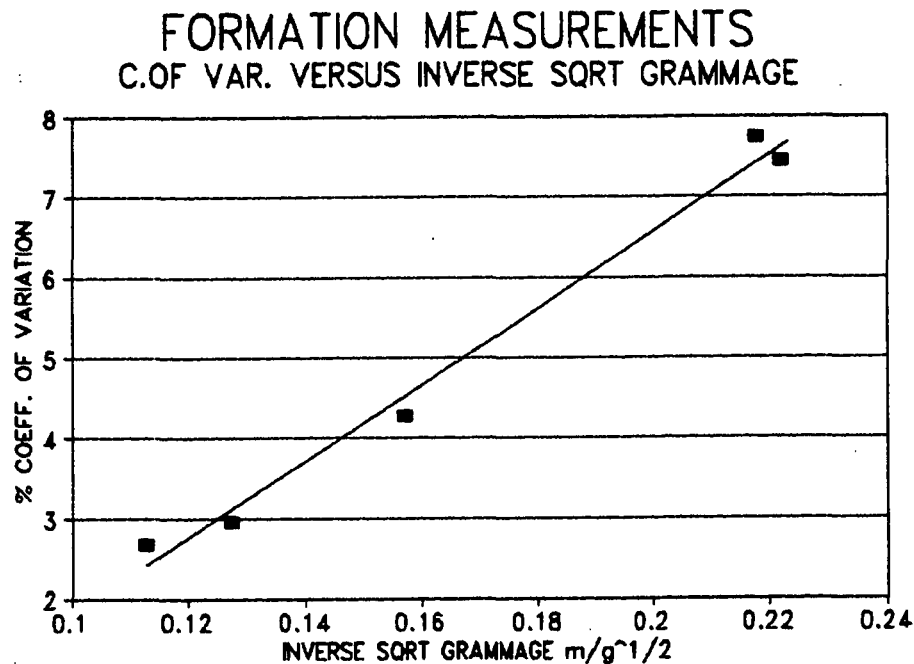


Figure 1.

If we are interested in visual uniformity it seems reasonable to use the coefficient of variation of optical density or its inverse. In other situations the variability in mass density may be more appropriate. Since most formation testers are based on light transmission measurements, the question is often asked as to how good an indication they give of mass density variability. It is well known that changes in refining, wet pressing and particularly calendering can result in significant changes in formation index when based on light transmission measurements, whereas mass density measurements may be relatively unaffected.

The relationship between optical density and grammage can be derived using the Kubelka-Munk equations. It can be shown that the optical density  $\ln(1/T)$  is related to the grammage  $W$  by the following equation:



$$\ln(1/T) = \ln[a/b \sinh b*s*W + \cosh b*s*W]$$

where  $T=I/I_0$  is the light transmittance,  $a$  and  $b$  are functions of sheet reflectivity  $R_{\infty}$ , and  $s$  is the scattering coefficient.

$$a = [1/R_{\infty} - R_{\infty}]/2$$

$$b = [1/R_{\infty} + R_{\infty}]/2$$

Clearly this will not yield a linear relationship between optical density and grammage as illustrated in Figure 2, where the measured variation of optical density (measured on the GERS) with grammage is compared with that predicted using the above equations and measurements of brightness and opacity (measured on the TB1). Also included in this figure are average measurements of optical density measured on the IPST Formation Tester. In this case we see that the optical density measurements are higher and again non-linear. The difference in level may be due to the fact that white light was used for the IPST Formation Tester measurements, whereas monochromatic light at 457nm was used in the GERS and TB1 instruments.

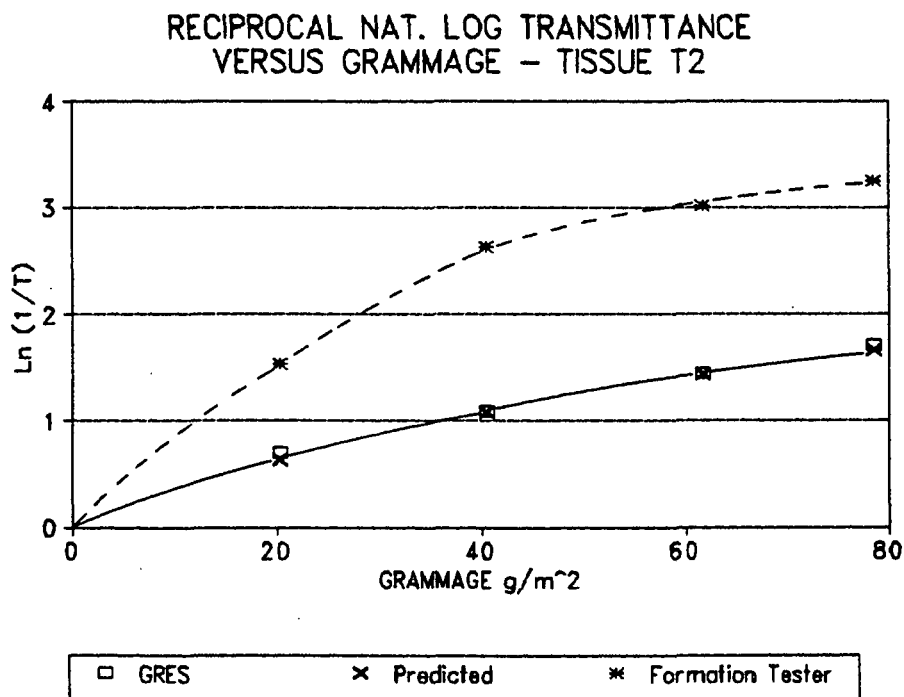


Figure 2.

To overcome the problem of non-linearity, it is proposed to assume local linearity. The slope B is then calculated by differentiating the above equation and using optical properties measured at that mean grammage level  $W_m$ . The relationship to predict the coefficient of variation of mass density from the coefficient of variation of optical density is then given by:

$$C.V.(W) = C.V.(\ln I/T)/(1 + A/W_m * B)$$

where

$$\ln(1/T) = A + B * W_m$$

The prediction of  $C.V.(W)$  from optical density measurements is compared with measured  $C.V.(W)$  in Figure 3. The agreement is not particularly good, but is perhaps reasonable considering the assumptions which have been made.

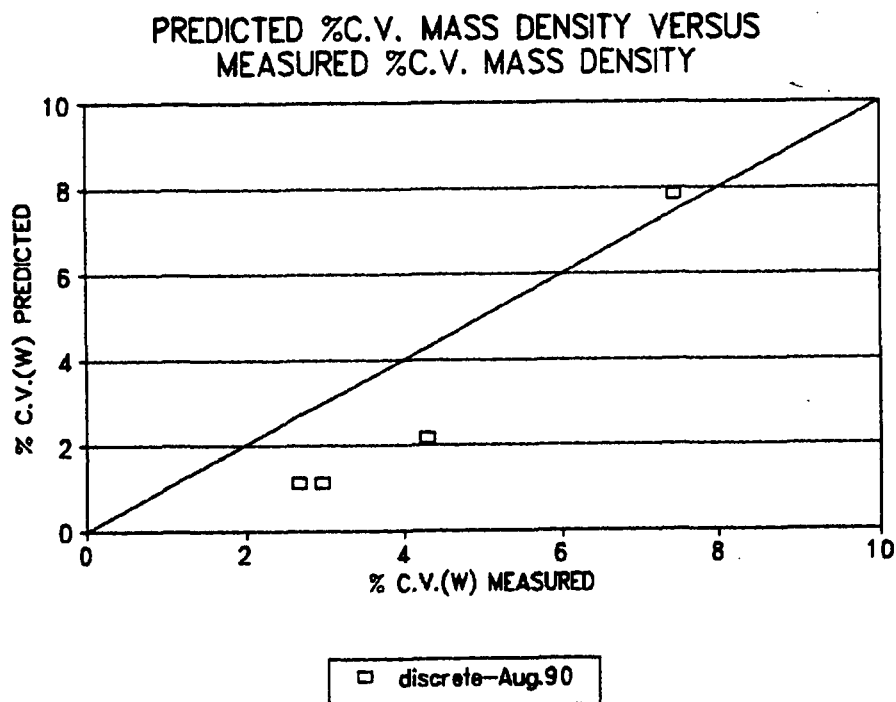


Figure 3.

### Effective Aperture Size

The geometric aperture of the IPST Formation Tester is 1mm x 1mm, and our measuring area for formation is typically 80mm x 80mm. A formation index based on the coefficient of variation, i.e. %C.V. is expected to vary as the inverse of the aperture area (3). Software has been written to calculate the %C.V. for different aperture sizes. The variation of %C.V. with aperture area for a Formette tissue handsheet is shown in Figure 4.

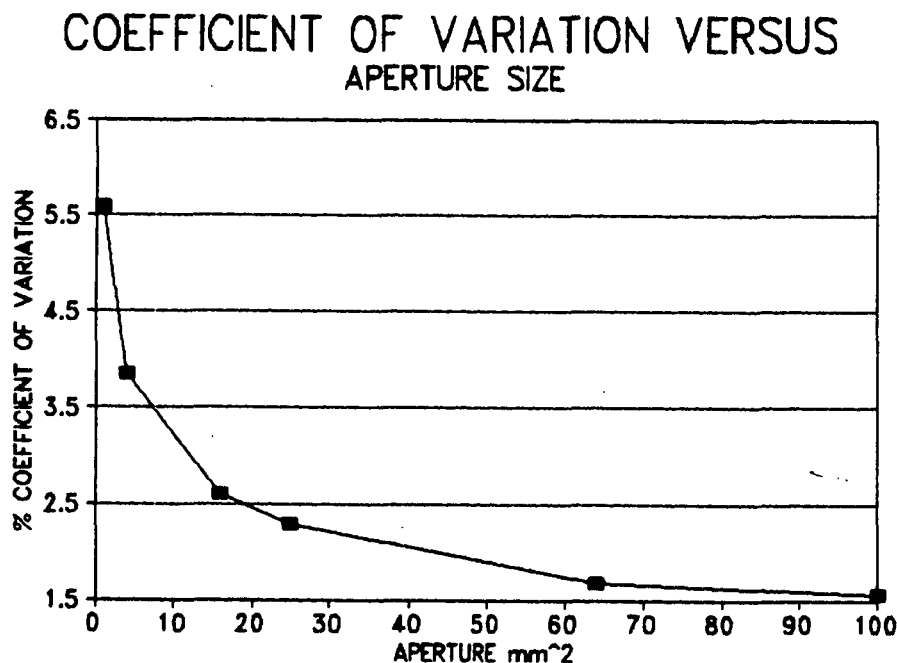


Figure 4.

### Image Analysis - Tracor Northern Image Analyzer

However useful %C.V. is as an index of formation, it is believed that it will not satisfy all of our needs for measures of formation for various converting and end use requirements. It has been shown by Cresson, T. (4) that sheets with the same %C.V. can have quite different distributions of mass and optical density.

We are particularly interested in applying image analysis and pattern recognition algorithms, e.g. Spatial Gray Level Dependence Method (4) to better characterize the formation of paper and board.

With respect to image analysis we have recently been able to input our formation data to the Tracor Northern Image Analyzer. Figures 5 through 12 show examples of various outputs from this system for two newsprint samples with large differences in their %C.V. The images are for both optical and mass density measurements on both samples.

#### Strength Dependence on Formation

Optical and mass density measurements are being made on tensile and fracture toughness samples both before and after failure.

#### COMBINED STRESS MEASUREMENTS

Examples of converting and end use performance areas where combined stresses are considered to be important include: corrugating, calendering, adhesive joints, and paper tube performance.

An out-of-plane biaxial device for measuring the failure envelope of paper and board when subjected to combined out-of-plane stresses is shown in Figure 13. We have now completed an investigation to determine the effects of refining and wet pressing on the out-of-plane failure envelope.

For this study oriented handsheets were made on the Formette Dynamique from a linerboard furnish, and dried on the IPST combined wet pressing and drying equipment to ensure fully restrained drying after pressing.

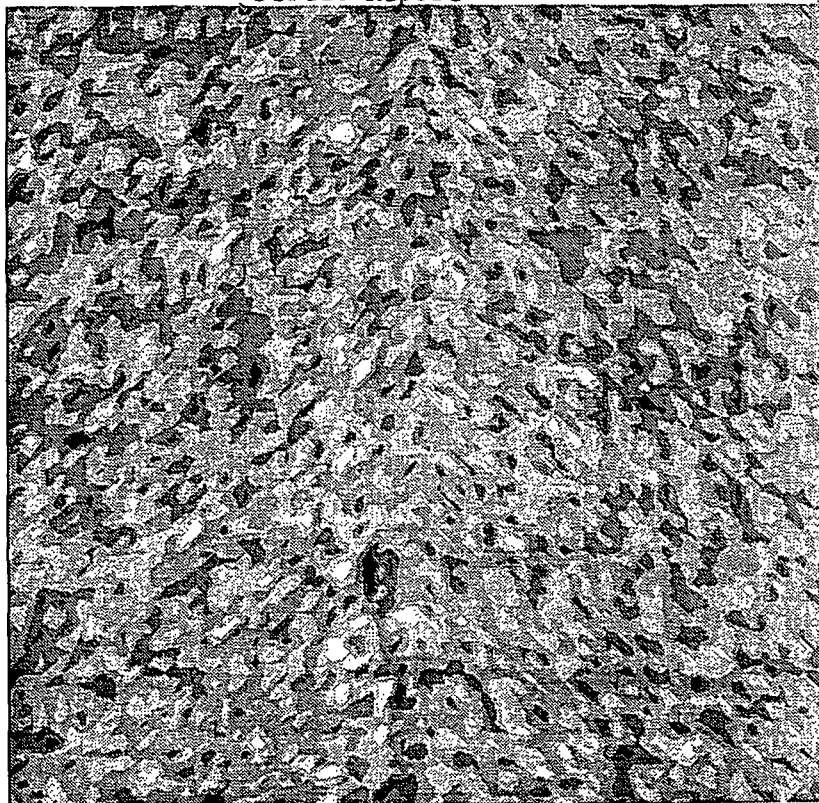


Figure 5 Contour Map Eight Gray Levels - Formette Handsheet  
(Newsprint) Transmitted Light C.V. (T) = 4.48%

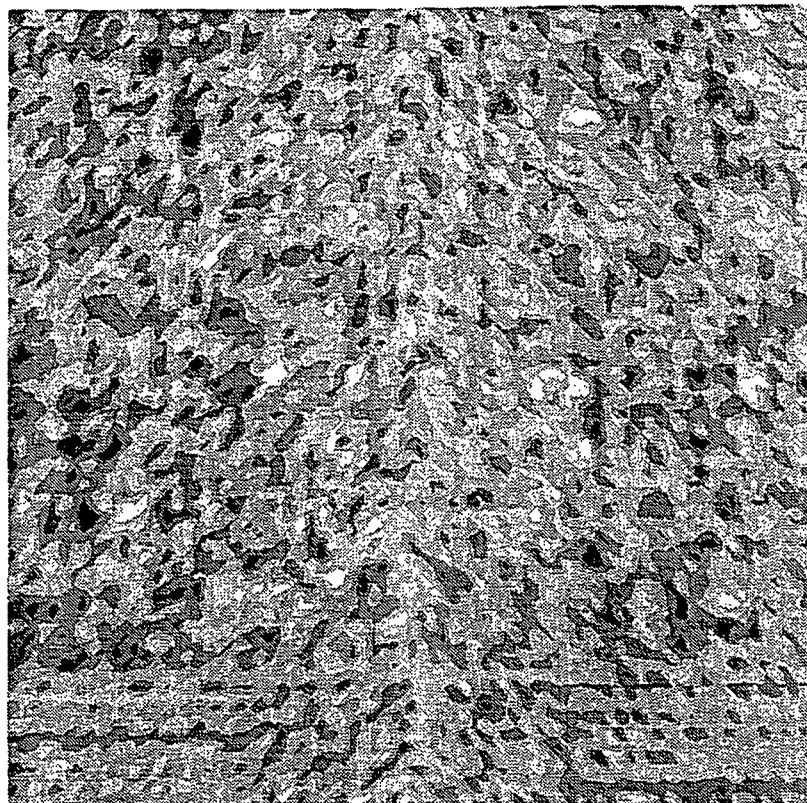


Figure 6 Contour Map Eight Gray Levels - Formette Handsheet  
(Newsprint) Beta C.V. (W) = 5.25%

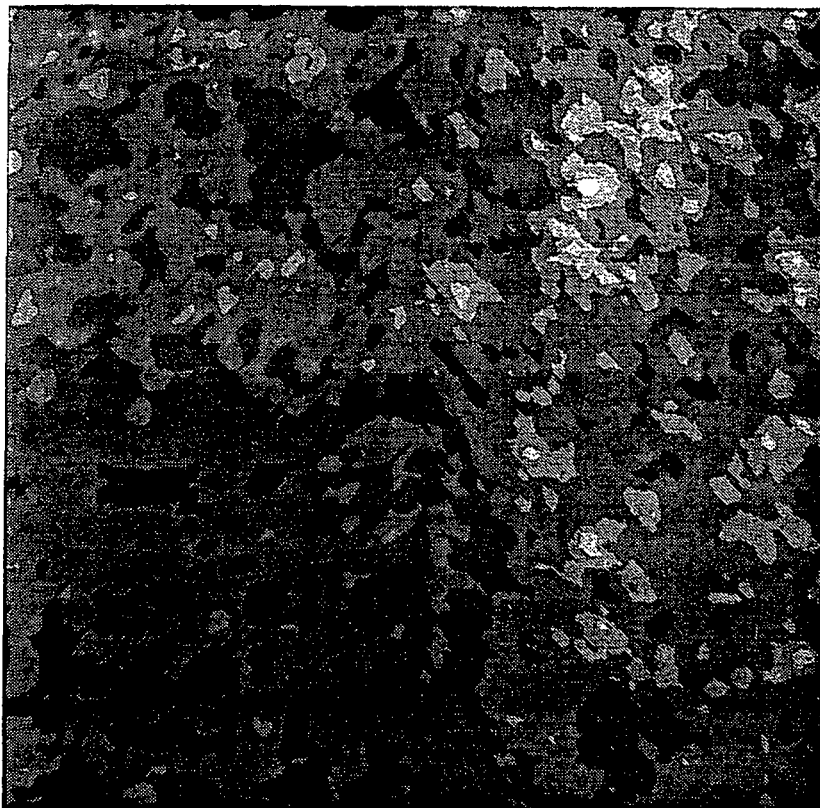


Figure 7 Contour Map Eight Gray Levels - N & W Handsheet  
(Newsprint) Transmitted Light C.V.(T) = 13.5%

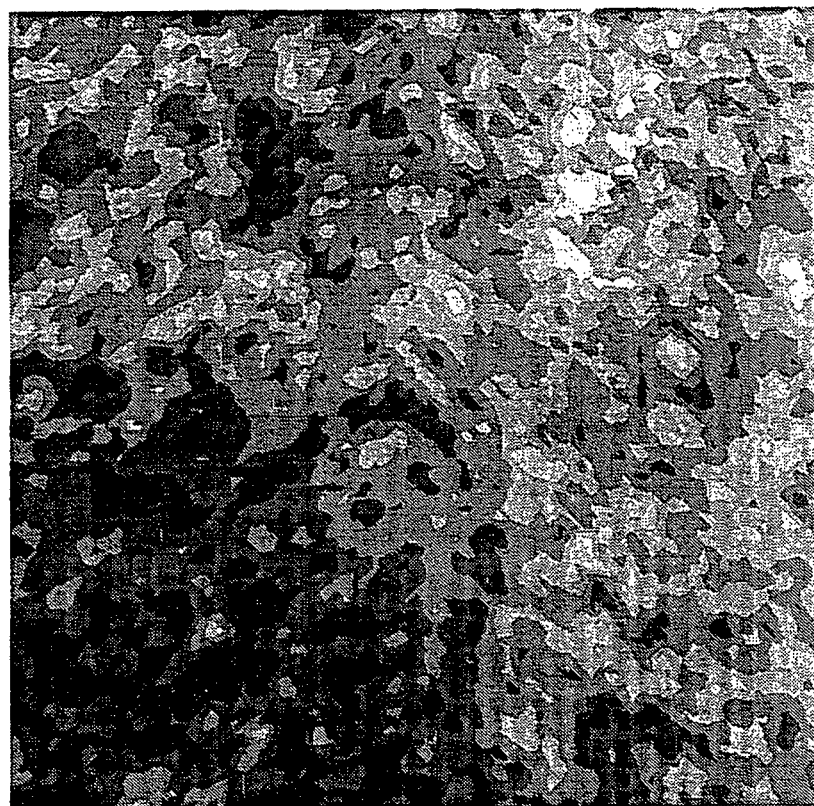


Figure 8 Contour Map Eight Gray Levels - N & W Handsheet  
(Newsprint) C.V.(W) = 19.03%

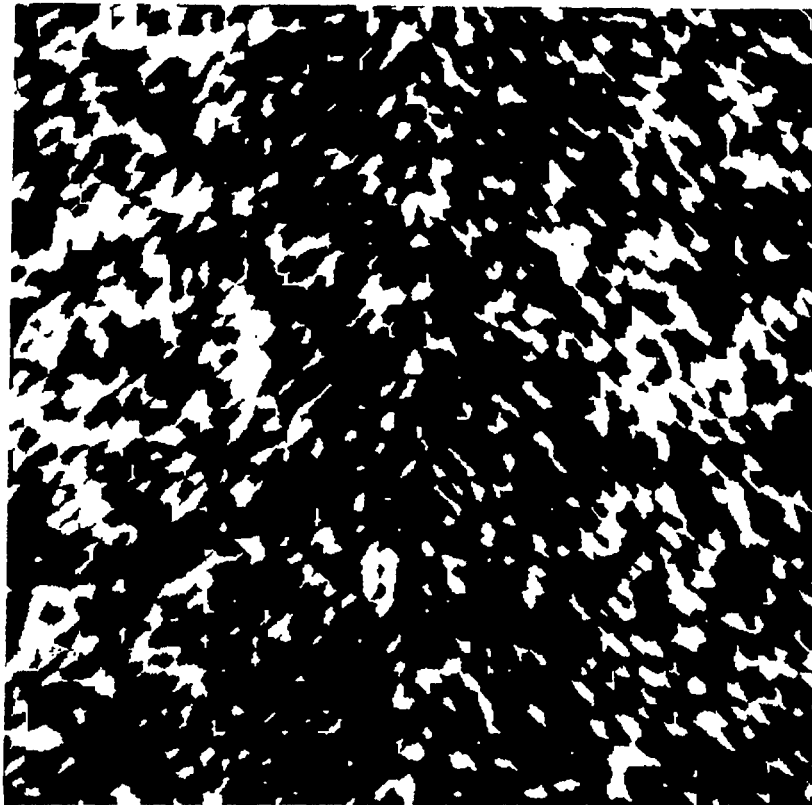


Figure 9 Binary Image Map - Formette Handsheet  
(Newsprint) Transmitted Light C.V. (T) = 4.48%

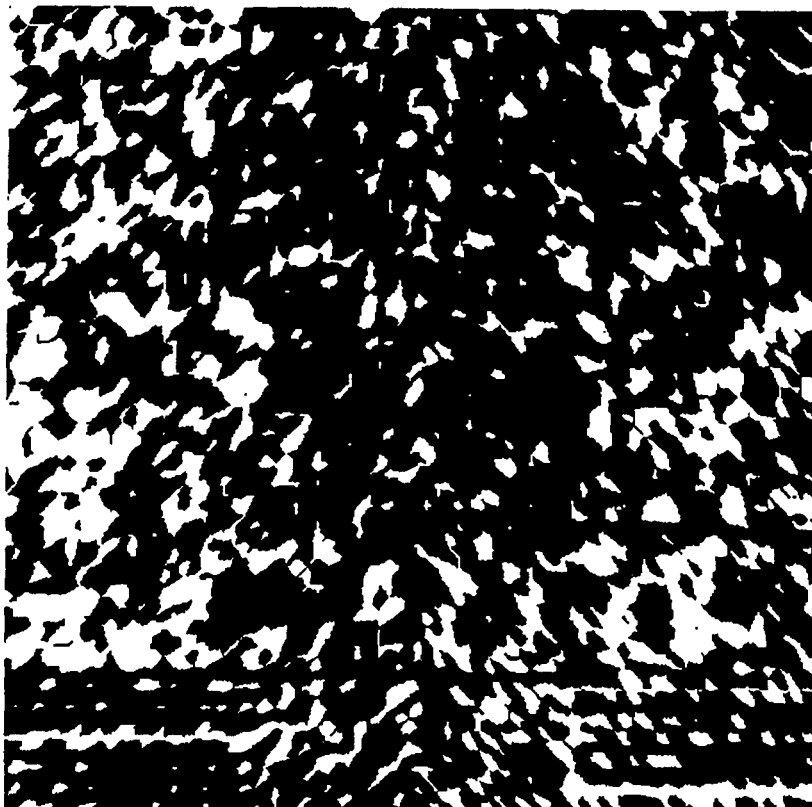


Figure 10 Binary Image Map - Formette Handsheet  
(Newsprint) Beta C.V. (W) = 5.25%

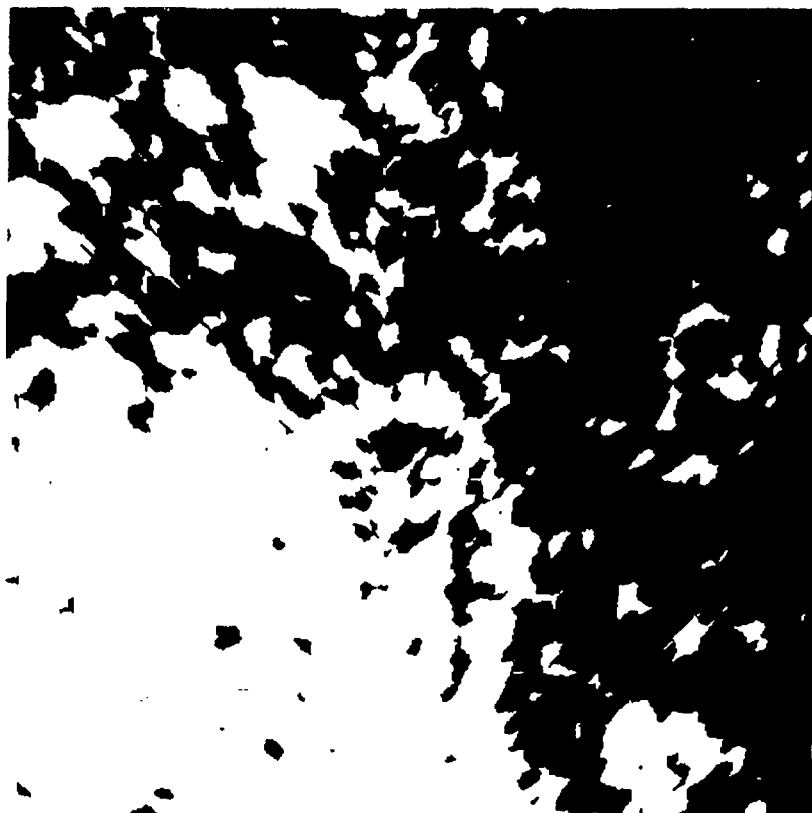


Figure 11 Binary Image Map - N & W Handsheet  
(Newsprint) Transmitted Light C.V. (T) = 13.5%

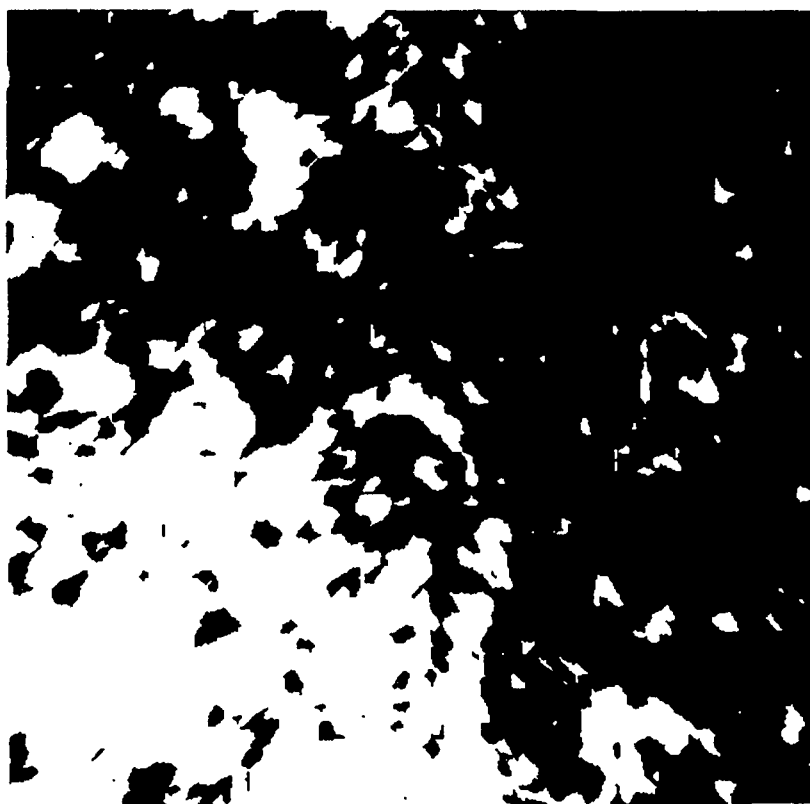


Figure 12 Binary Image Map - N & W Handsheet  
(Newsprint) C.V. (W) = 19.03%



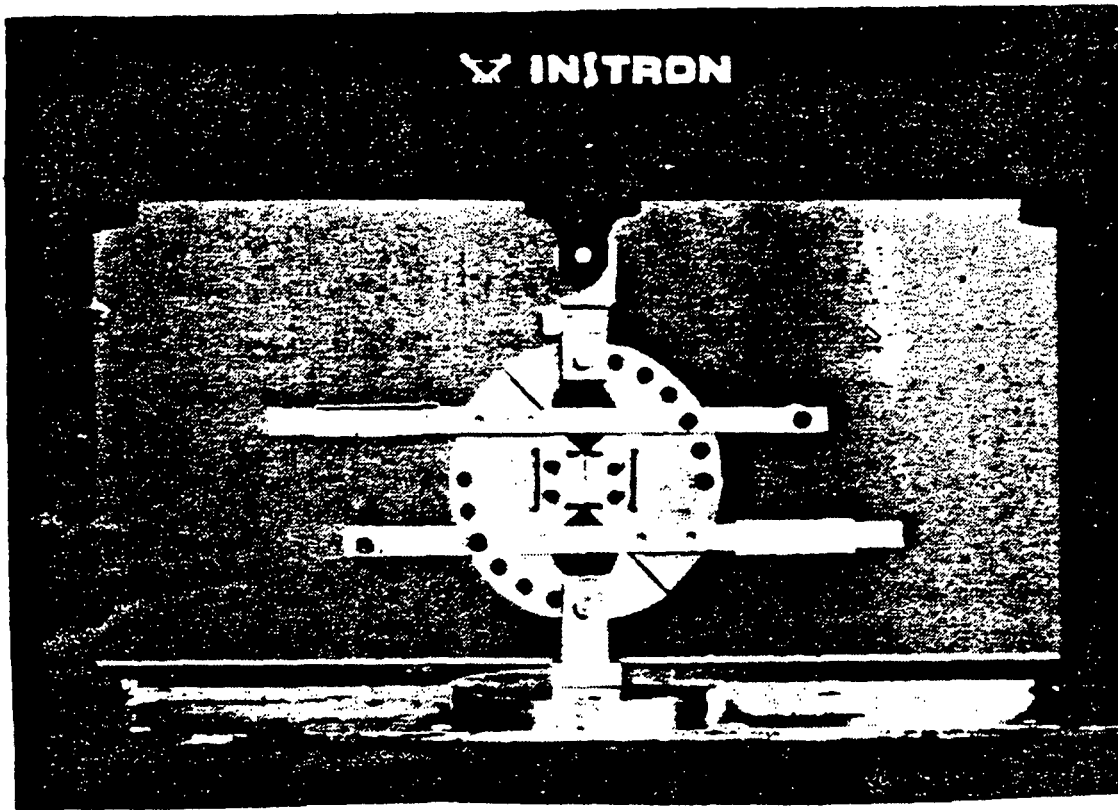


Figure 13.

Ultrasonic non-destructive characterization of the Formette handsheets, and the samples (1.5" x 0.5") which were subsequently subjected to biaxial testing is shown in Figures 14 through 18.

Figure 14 shows an envelope for the in-plane elastic properties, while the out-of-plane properties for wet pressing and refining collapse on to a common regression line, i.e. Figures 15 and 16. As shown in Figures 17 and 18 the test samples exhibit considerably more variability in out-of-plane properties, particularly the out-of-plane shear constant.

The variation in failure stress from pure shear in 15 degree increments to pure normal stress is shown in Figures 19 through 24. It appears that the failure stress-apparent density variation is independent of whether the density level was achieved by refining or wet pressing, which is consistent with the elastic behavior.

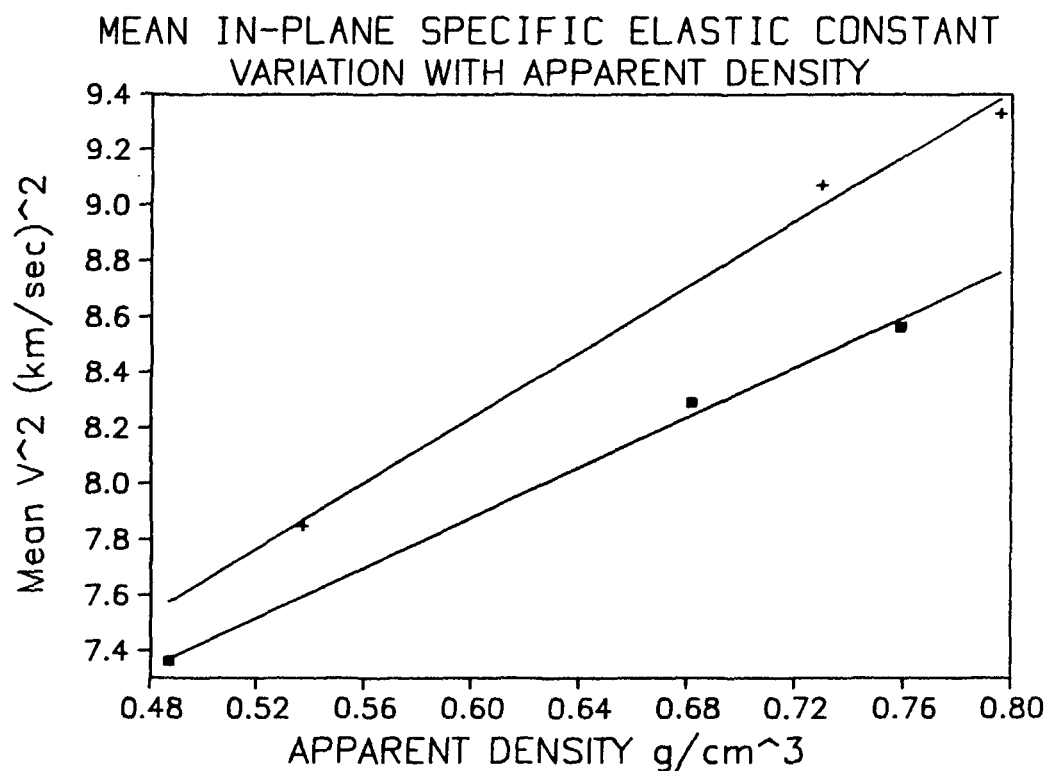


Figure 14.

LONGITUDINAL OUT-OF-PLANE MODULUS  
VERSUS APPARENT DENSITY

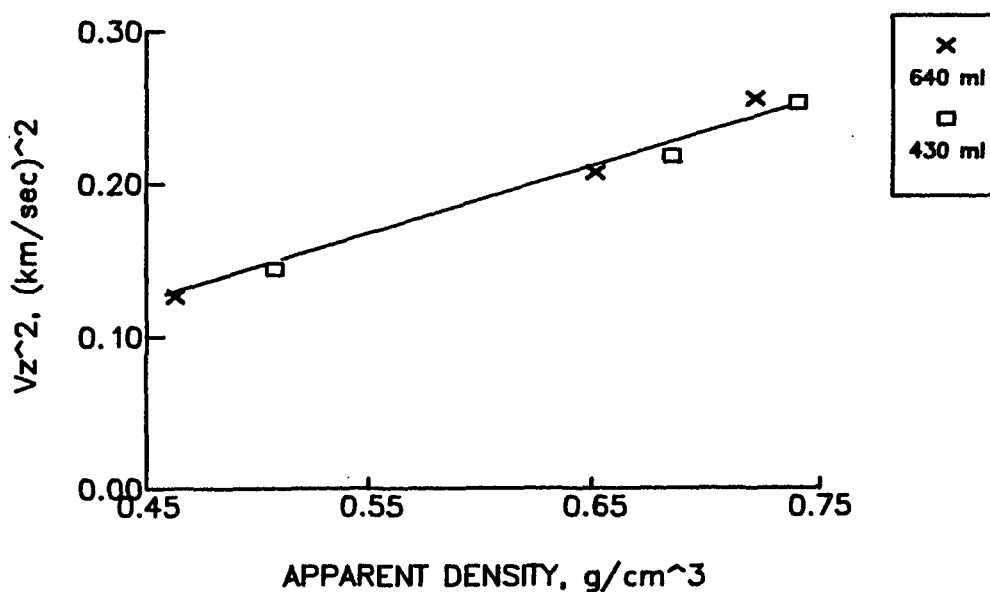


Figure 15.

### MD & CD OUT-OF-PLANE SHEAR MODULI VERSUS APPARENT DENSITY

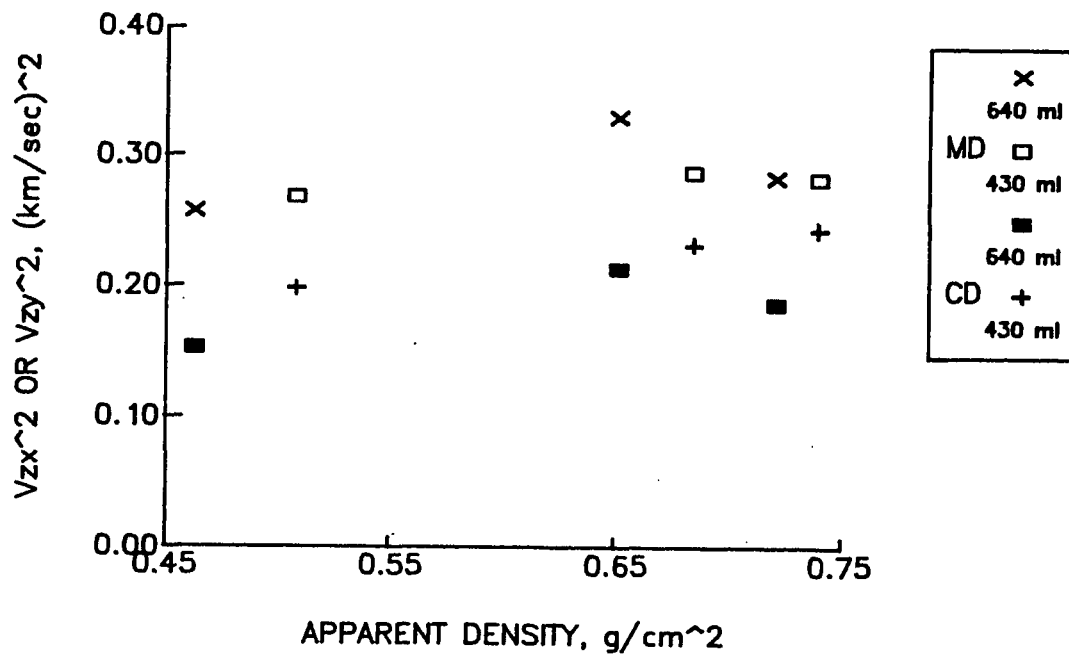


Figure 16.

### COMBINED STRESS MEASUREMENTS Out-of-Plane Elastic Constant

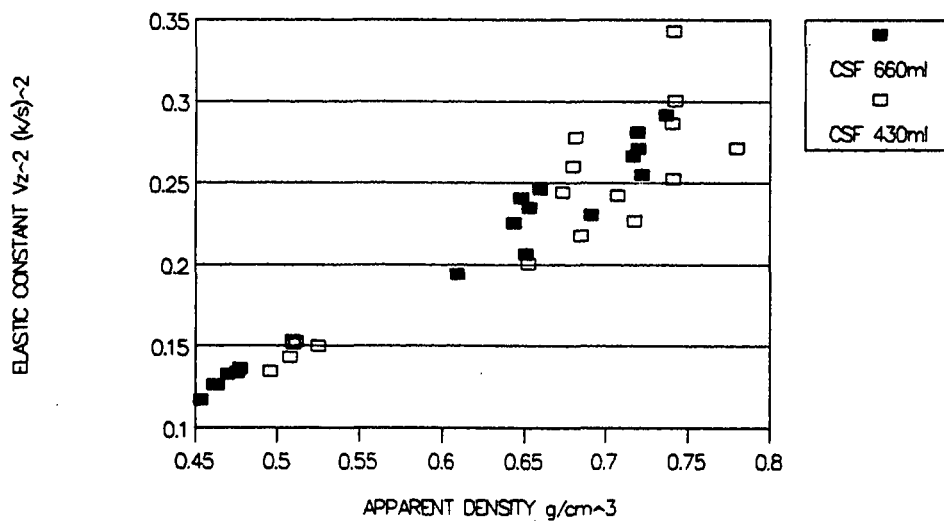


Figure 17.

# COMBINED STRESS MEASUREMENTS

## MD Out-of-Plane Shear Elastic Constant

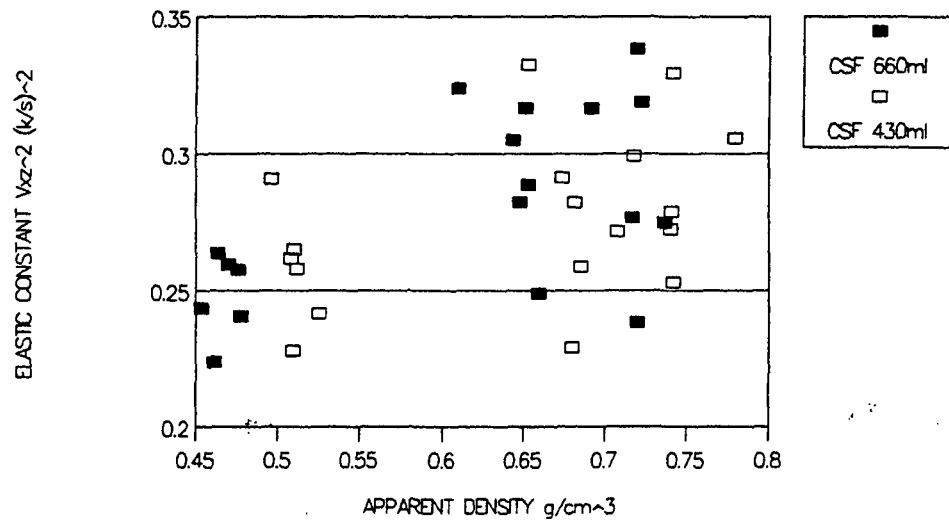


Figure 18.

From Figures 19 through 24 envelopes have been constructed for three density levels, i.e. 0.5g/cm<sup>3</sup>, 0.6g/cm<sup>3</sup>, and 0.7g/cm<sup>3</sup> and are shown in Figure 25. The solid lines are theoretical predictions based on the work of Liu, J. (5), which he used to analyze the in-plane biaxial failure envelope of wood coupons.

$$\sigma_f(\theta)^2 = \frac{\sigma_s^2 * \sigma_n^2}{\sigma_s^2 * (\cos \theta)^2 + \sigma_n^2 * (\sin \theta)^2}$$

where  $\sigma_f(\theta)$ , is the failure stress at angle  $\theta$  from the pure normal stress axis and  $\sigma_s$ ,  $\sigma_n$ , are the pure shear and normal failure stresses respectively. The agreement appears to be quite good despite the high level of variability which can arise in these types of measurements.

A paper based on this research has been submitted and accepted for presentation at the International Paper Physics Conference to be held in Hawaii this September.

#### STUDENT RELATED WORK

Last January Thomas Bither successfully defended his Ph.D thesis "Strength Development by Refining and Wet Pressing" A joint paper based on this research has been submitted and accepted for presentation at the International Paper Physics Conference to be held in Hawaii this September.

### COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - Pure Shear

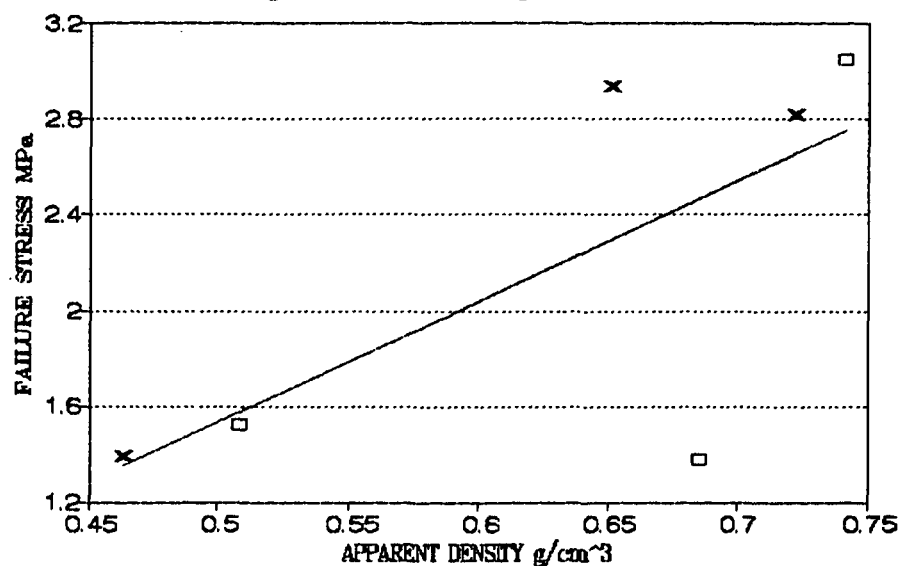


Figure 19.

### COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - 75 Degrees

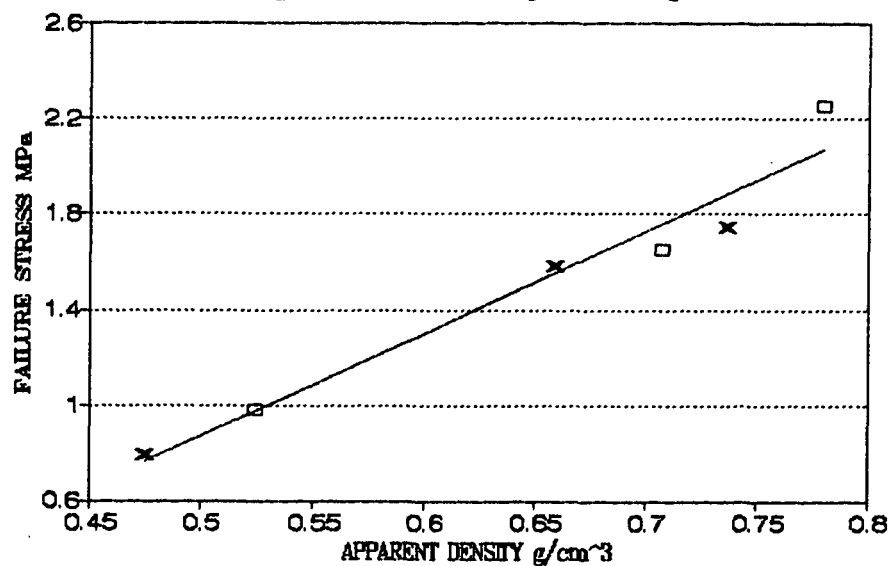


Figure 20.

### COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - 60 Degrees

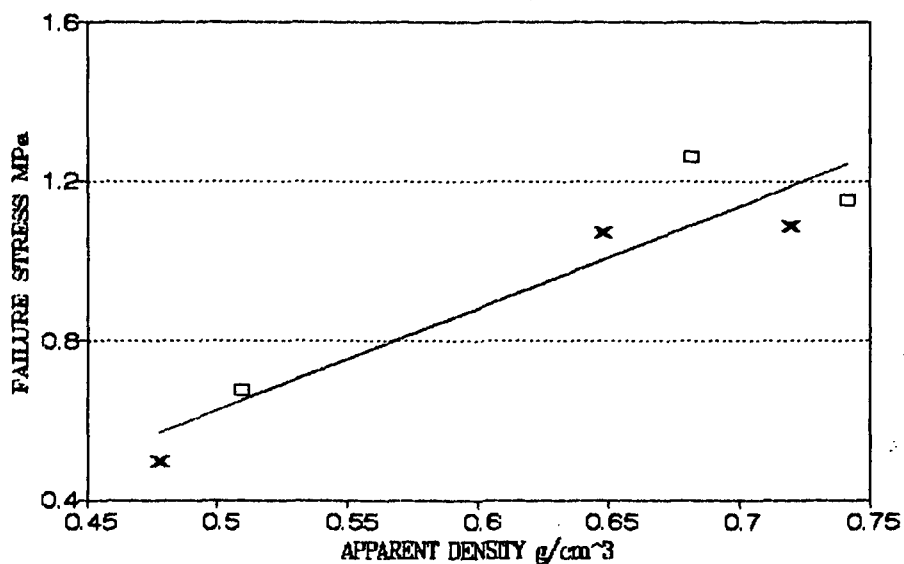


Figure 21.

### COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - 45 Degrees

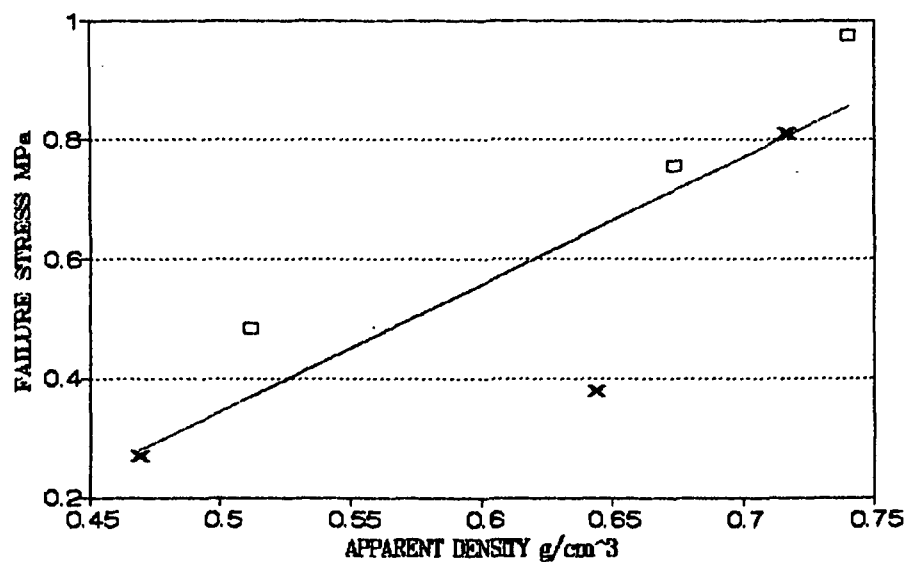


Figure 22.

## COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - 30 Degrees

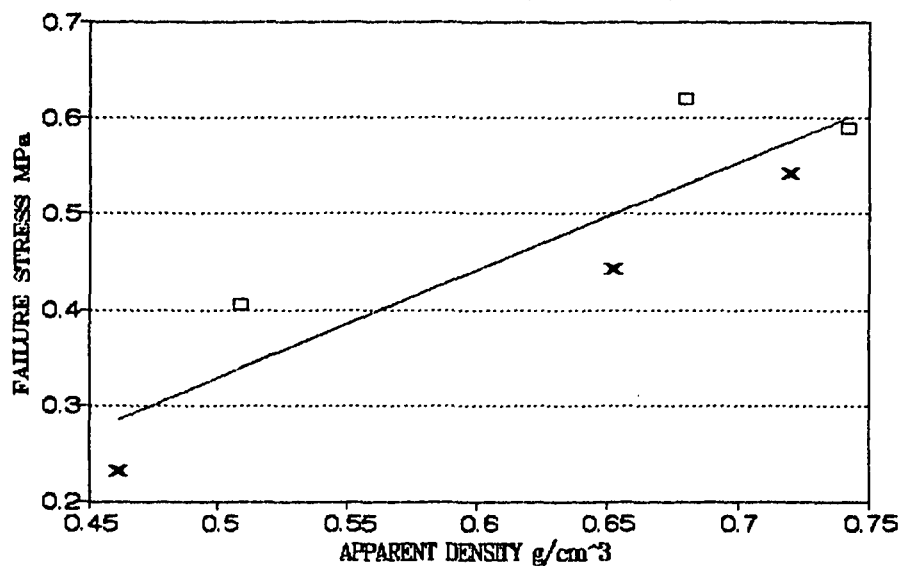


Figure 23.

## COMBINED STRESS MEASUREMENTS

Refining and Wet Pressing - Pure Z

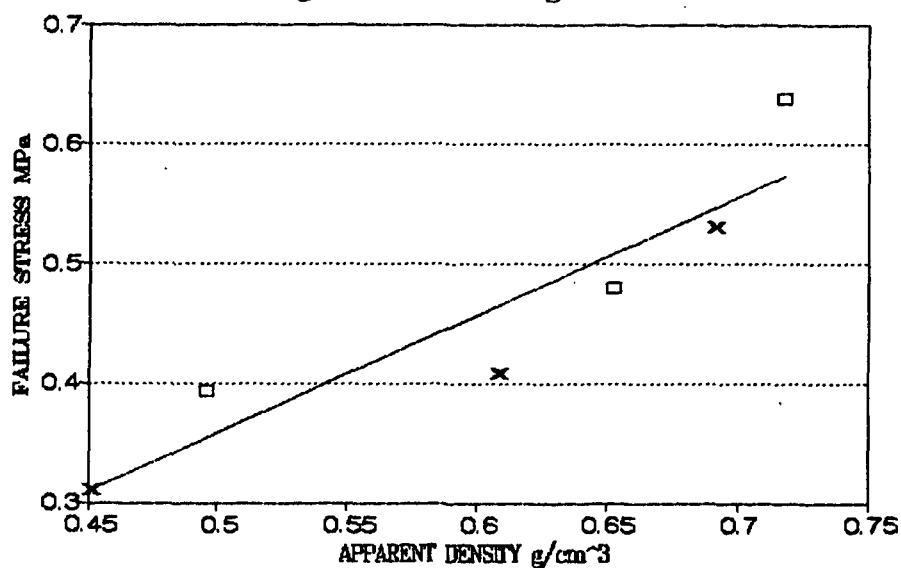


Figure 24.



## COMBINED STRESS MEASUREMENTS

## Refining and Wet Pressing

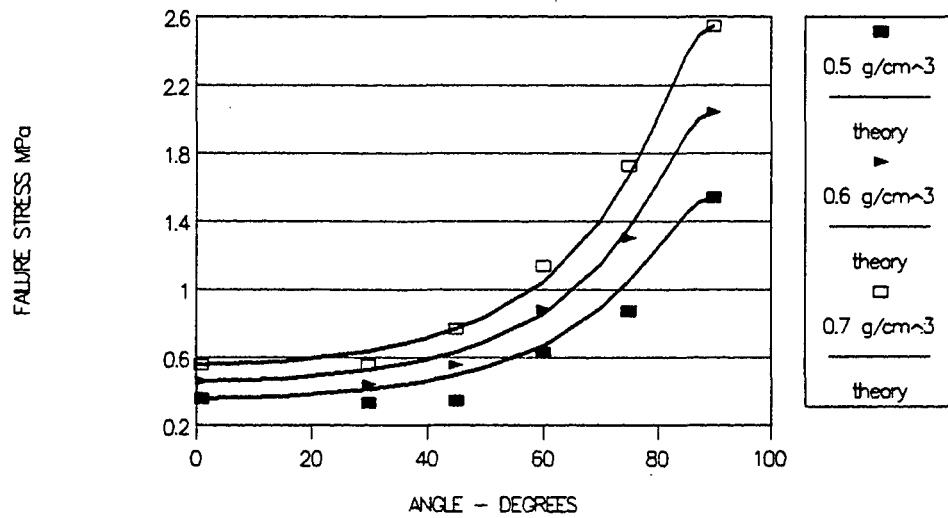


Figure 25..

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5. Liu. J.

## 1. COMPRESSIVE STRENGTH IMPROVEMENT

## A Progress Report

## SUMMARY

This investigation is concerned with how raw materials, papermaking process variables, and converting operations might be better used to evolve strategies for the improvement of compressive strength.

In recent years a flurry of activity has been directed toward techniques for measuring compressive strength, as well as understanding the mechanisms of failure, and the development of models and equations for its prediction. Our research has been guided mainly by the compressive strength model developed by Habeger and Whitsitt, which demonstrates that compressive strength is dependent on both in-plane and out-of-plane elastic properties. These elastic properties are in turn dependent on raw materials selection and the papermaking process.

## RAW MATERIALS

It is evident from the literature that fibers with high elastic constant values have low fibril angle and are defect free, i.e., devoid of kinks, curls, and microcompressions. There are, therefore, opportunities in fiber selection to maximize fiber compressive strength potential and stock treatment either to avoid or to remove fiber defects.

It has been demonstrated that a 52% yield pulp may not necessarily represent an optimal yield level for maximizing specific compressive strength. However, if higher yield pulps are to be effectively employed, some form of "softening" treatment may be required, i.e., sulfonation, press drying.

A technique has also been developed for directly measuring the compressive strength potential of 16mm x 16mm wood coupons (or veneers) and directly relating it to the compressive strength measured on mini-handsheets made from the fibers comprising the wood coupon.

A number of polymer systems have been investigated to determine their effect on compressive strength. In student-related work it was found that a significant improvement in compressive strength could be obtained with dry web addition of

polyvinyl acetate, presumed to be mainly interfiber reinforcement. Attempts to reinforce the fiber cell wall with polyvinyl acetate via solvent exchange were not successful. The furnish addition of poly(aminoamide) epichlorohydrin (PAE) resin led to a significant improvement in compressive strength. Furthermore, it was found that its effectiveness improved with increased wet pressing. Combination of various starches with PAE did not produce any marked synergistic effects; however, cationic starch addition did produce a significant improvement in compressive strength. The Formette has proven to be a useful tool in evaluating additives, particularly with respect to measuring the relative retention of unbleached pulp fines and starch. This approach may be helpful in looking at more realistic mill white water systems, i.e., addressing the problem of cationic demand.

## PAPERMAKING

The papermaking process offers a number of opportunities for improving compressive strength. We have confirmed the findings of other researchers that refining is limited in its effectiveness to improve compressive strength. Furthermore, we believe that the Habeger-Whitsitt model is useful in explaining this effect, i.e., only the in-plane elastic constants are differently affected by refining and wet pressing, but compressive strength is only dependent on the in-plane modulus raised to the three-quarter power, i.e.,  $(E/\rho)^{0.75}$ .

Although impractical, we have demonstrated that sheets made from 100% "fines" can exceed compressive strength plateau levels produced by more conventional refining and wet pressing.

Forming consistency was changed in the range of 0.5% to 2.0% using Noble and Woods and the Formette Dynamique sheet formers. Compressive strength was not sensitive to forming consistency, although a significant loss in tensile strength of the Noble and Woods handsheets was found. No losses in tensile strength for the Formette sheets was found over the same consistency range. Unexpectedly, no significant increase in longitudinal elastic modulus was found with increasing forming consistency for either former.

In exploratory work, we have found that the type of wet press felt can have a significant impact on properties including compressive strength. Generally,

commercial felts result in a lower compressive strength than can be obtained using either blotters or a porous plate.

The importance of drying restraint in obtaining high levels of elastic constants and compressive strength cannot be overemphasized. We have also found under conditions of full restraint that as wet pressing is increased, the elastic anisotropy of the sheet is reduced. Therefore, techniques to improve CD drying restraint are an obvious need.

## CONVERTING

Converting operations such as calendering and corrugating of medium are potential sources of compressive strength losses. In limited calendering experiments, losses in elastic properties and compressive strength have been measured. Precise reasons for these losses have yet to be found, but include the possibilities of "damage" and the relaxation of internal stresses. Calendering strategies, e.g., gradient calendering, are therefore needed to ensure these losses are minimized. Improved wet-end control may also reduce the need to take corrective action at the calender stack. A loss in compressive strength (forming loss) can also occur during the corrugating of medium. It is speculated that if the interlaminar shear resistance of the medium can be lowered during papermaking and restored during corrugating then these losses might be reduced.

Although excellent correlations have been found between compressive strength and elastic properties using the simplified Habeger-Whitsitt model, i.e.  $\sigma_c/\rho = (E/\rho)^{0.75} \cdot (E_z/\rho)^{0.25}$ , the correlation changes with pulp type, additive addition, and calendering. Further work is therefore required to determine the reasons for these changes.

BOARD PROPERTIES AND PERFORMANCE

STATUS REPORT

FOR

PROJECT 3571

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

April 4, 1991

## PROJECT SUMMARY

Date: April 1, 1991  
Project : BOARD PROPERTIES AND PERFORMANCE  
Project Code: BOARD  
Project Number: 3571  
Budget: \$100,000  
Project Staff: M.P. Sarfarazi

### PRIMARY AREA OF INDUSTRY NEEDS:

Converting/ Corrugating of Paper into Paperboard

### PROGRAM GOAL:

Develop an understanding of the mechanics and micromechanism of damage and failure commonly evolved during converting processes in corrugating mediums and other paper products.

### PROGRAM OBJECTIVES:

To ultimately optimize the machine design and operating conditions in order to minimize damage during converting and, therefore, improve the performance/cost ratio of medium and paperboard. The fundamental objectives are: (1) to study the mechanics of corrugation and the stresses developed during the flute formation process (2) to study the mechanism of flute damage process (3) to determine how the operating conditions such as nip temperature and moisture content and corrugating speed (characterizing the strain-rate) may be changed in order to minimize damage (4) to investigate and rationalize the effect of geometry such as caliper and flute radius on damage.

## PROJECT RATIONALE:

It is well known that a fluted medium exhibits much lower strengths than its uncorrugated counterpart. Previous work at the Institute has indicated that the compressive strength of a corrugating medium may be reduced by as much as 42% due to the fluting process, which in turn, may reduce the ECT of the board. The results of the short span MD tensile test on the formed medium has also shown reductions in the tensile strength when compared to the uncorrugated medium. This reduction in tensile strength is, however, somewhat less when compared with compressive strength. Forming can also reduce the ECT potential of the medium in the CD, which may be typically in the 20-30% range. The reductions in the compressive and tensile strengths in the MD and CD are due to the evolution of diversified micromechanisms of damage (e.g. disbonding, delaminations, fiber microbuckling, fiber fracture), which occur as a result of various types of stresses inflicted on the medium during the fluting process.

At the entrance to the labyrinth, the main stress on the medium is due to the applied brake tension, which may be considered to be approximately constant over the medium thickness. In the labyrinth, the medium is also under the influence of a frictional drag, which progressively increases the web tension as the medium is formed into a fluted profile. Previous experiments have indicated that increasing the coefficient of friction from 0.2 to 0.3 causes a 100% increase in the final tension of the medium. The results of the runnability studies in the past have indicated that the web tension increases and, therefore, the tensile strength decreases gradually as the speed of corrugation is increased (reflecting the effect of strain rate). Substantial increases in the web tension may also result due to vibration. During the fluting process, bending may also inflict significant tensile as well as compressive stresses in the medium. If the overall stresses induced in the medium are too high under the prevailing conditions of heat, moisture and rate of stressing, flute fracture may occur. At lower stresses, although cracks or defects may not be visually evident, microstructural damage can severely reduce the strength potentials of the medium. On the other hand, high temperature and moisture content have a plasticizing effect on the medium and reduce its tendency for damage and brittle fracture during fluting. It is expected that a medium is more prone to damage and fracture at low than high rates of loading. Of fundamental interest is, therefore, the study of the



**micromechanism of damage and failure under direct tension in corrugating mediums and related paper products at various conditions of temperature, moisture content and strain rate.**

Although uniaxial tensile forces are significantly responsible for inflicting microstructural damage to the fibrous network of a corrugating medium, there are other types of stresses which are commonly developed in the medium during the fluting operation. For example, during the fluting process, bending generates compressive as well as tensile stresses in the medium. If the compressive stresses in the medium are very high, microbuckling of individual fibers can occur, which is a significant mode of damage and, therefore, can seriously reduce the compressive strength potential of the medium and the associated corrugated board. **The study of the microbuckling phenomenon of individual fibers due to excessive compressive stresses, therefore, has sufficient relevance in the micromechanics of fluting.**

The flank/tip regions are particularly critical when considering the flat crush strength of the combined board. Of primary importance is **a comprehensive study of the stresses developed in the medium during the fluting process.** Since paper is a relatively thin structure and is, therefore, highly flexible, the finite element analysis should involve consideration of non-linear theories of continuum mechanics. Generally, the bending stresses and strains depend on medium caliper as well as the radius of curvature of the corrugating roll flute tip. The higher the caliper or the smaller the radius, the larger the strain and the likelihood of damage and fracture. In order to be realistic, the effect of strength losses due to damage (which occurs as a result of excessive tensile web stretch) should also be incorporated in the analysis. **Laboratory study should be conducted in order to determine the location, the extent, and the mode of damage to the medium during the fluting process.**

Formation of flute cannot occur through pure bending as there are always shear stresses present in the medium. Shear stresses in the medium reduce the net bending strain and, therefore, allow forming without excessive flute damage or fracture. If the elastic properties of the medium are such that it is significantly stiffer in bending than shear, flute formation could generate large shear strains. Since due

to the layered nature of the medium its transverse moduli is often quite low when compared with its in-plane moduli, the excessively high shear strains may cause delamination during corrugating. Therefore, of interest in the study of the micromechanics of paper damage and failure during the corrugation process is a **fundamental understanding of the problem of delamination of paperboard under the influence of excessive shear.**

#### PLANNED ACTIVITIES FOR THE FISCAL YEAR 1991-1992

During the upcoming year we will initiate a new program of study related to the micromechanics of damage and failure in paper and paperboard. This is primarily motivated by the fact that during converting processes these products often experience severe damage and loss of strength. It has already been emphasized that a number of fundamental issues are indigenous to the problem of paper damage during the converting processes. These include:

1. Damage and failure of a fibrous network structure under direct tension.
2. Problem of microbuckling of individual fibers under compression.
3. Shear delamination of paperboard and related laminates.

In the coming year we will primarily focus on the problem of damage and failure of paper under the influence of tensile stresses. Of interest is the study of the non-linear constitutive behavior of paper when it is subjected to progressively damaging uniaxial tensile stresses. The first objective is to observe the fibrous network structure under microscope as the sample is being progressively damaged, and to identify the exact modes of damage (disbonds, fiber fracture, voids, etc.) occurring during the tensile straining process. Various microscopic techniques will be explored including:

- a. Scanning electron microscopy
- b. Transmission electron microscopy

- c. Scanning laser microscopy.
- d. Scanning laser acoustic microscopy
- e. Scanning acoustic microscopy

Priority will be given to the scanning electron and the scanning laser microscopy techniques. The Institute is currently in the process of acquiring a new scanning electron microscope. A tension device needs to be mounted on the new SEM in order to be able to stretch a sample continuously and monitor its damage progressively. The scanning laser microscope (SLM) has been successfully applied to non-destructive evaluation of semi-conductor materials and devices in the past. Its operation is based upon the concept of confocal optics which uses the same objective lens for illumination and reflection. Capable of real-time imaging, it is indispensable for observing samples that change their morphology quickly such as one containing a fast propagating crack. It is particularly advantageous in the field of pulp and paper research because contrary to SEM, no pre-treatment of the sample is required. On the other hand, acoustic microscopy requires the use of a fluid couplant which is often polar in nature and, therefore, disintegrates the fibrous structure of the paper during sample observation.

Another objective of the aforementioned research program will be to characterize and measure damage through a well-established non-destructive experimental technique. The primary candidate is the acoustic emission measurement technique. This method is based upon the fundamental idea that when the paper sample is damaged, a transient wave is emitted upon rapid release of strain energy from the microstructure. The current apparatus set-up is such that a resonant-type transducer is attached to the paper sample using a paper clip or via a couplant such as silicone grease. The transducer contains a ceramic plate which is very sensitive to the acoustic emission of ultrasonic wave. The paper sample is progressively damaged through continuous tensile straining using an Instron Machine. The signal from the transducer is preamplified and directed to an acoustic emission data analyzer. The signal is filtered through a high-pass filter with a low cutoff frequency of a certain threshold in order to eliminate noise. The counts, amplitude and various

other standard acoustic emission measurements are recorded. The list of data also includes the strain and load signals from the Instron Machine. The initial exploratory acoustic emission experiments conducted at the Institute have been quite encouraging. It seems that the acoustic emission technique characterizes damage in paper materials rather well. We, therefore, plan to continue with our acoustic emission studies of damage in paper in the future. We are currently in the process of buying an acoustic emission data analyzing equipment. We have been recently looking at the available products in the market and have found that the prime candidates for purchase are the Physical Acoustics Corporation's LOCAN AT and the Hartford Steam Boilers Inspection Technologies' AET 5500. Furthermore, we have decided that the former is to be preferred considering their costs and capabilities. Although we have had these equipments on consignments for a period of two weeks at a time and have, therefore, performed numerous tests, these experiments are to be considered as exploratory in nature and no formal presentation or documentation of the results will be carried out at this time.

Apart from the observational characterization and experimental measurement of damage we plan to analytically simulate the flute formation process using the finite element method and compute the typical medium stresses developed during corrugation. We are currently in the process of purchasing a 386 personal computer which is capable of housing the rather sophisticated finite element program COSMOS. The Institute had initially purchased an old version of this code for PC, but we understand from the representative of the company that new and updated versions of the software are now available which are more suitable for the complex non-linear type of modelling required by our analysis. We are planning to acquire the latest version of the FEM code COSMOS in the near future.

We also plan to conduct off-site laboratory experiments in which a sample of corrugating medium could be simultaneously stretched and bent about rods of well-defined radii in order to simulate the flute formation process. Microscopic and nondestructive evaluation techniques could be employed to assess the location, the type, and extent of damage developed during such a process.



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